Investigation on the Rheology of Dilute Partially Hydrolyzed Polyacrylamide Solution

by

Nor Asyiqin binti Zainal Abidin

Dissertation submitted in partial fulfilment
of the requirements for the
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(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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Approved by,

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(Mr. Mohd Zamri Abdullah)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

NOR ASYIQIN BINTI ZAINAL ABIDIN

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ABSTRACT

New procedures are presented and existing procedures are reviewed to aid in the measurement of properties related to performance of polyacrylamide solutions. Recently the research interest of partially hydrolyzed polyacrylamide (HPAM) is increasing because HPAM is broadly used in various industrial and commercial applications, especially in oil and gas industry. Oscillation measurement tests were conducted to characterize the viscosity behavior of HPAM. In this project paper, a survey is given on rheological properties of partially hydrolyzed polyacrylamide solution. Master solution of HPAM at 300, 500, and 700ppm and also diluted solutions which ranging from 10 to 50ppm are tested to find the rheological properties of HPAM. The result of hydration time and concentration of master and dilute HPAM were investigated as well. In order to accomplish this project, research on the different concentration and the hydration period will be conducted on how strength of polymer will affect the viscous and elastic modulus behavior. Both master and dilute polymer solutions will be analyzed using *Bohlin Gemini 2 II* rheometer at a closed room temperature of 25°C. These polymer solutions were performed to make a comparative analysis of the results and the modulus behavior profile are observed based on the hydration period and concentrations given.

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CHAPTER 1: INTRODUCTION

1.1 Background of Study

Polymer flooding is well-known technique belongs to enhanced oil recovery (EOR) with over 40 years of wellbeing application over a broad range of reservoir conditions. It is one of the chemical injection methods in which the polymer is dissolved in the injected water to increase the viscosity and enhance the efficiency of sweep in the hydrocarbon reservoir in order for the ease of pumping to the surface. A typical polymer flooding is involving mixing and injection of the polymer depending on the time until it reaches at least 30% of the pore volume of reservoir has been injected. The polymer is then continues to a long-term water flooding to brings out the polymer slug and the oil bank in front of it towards the production wells ("Enhanced Oil Recovery," 2013).

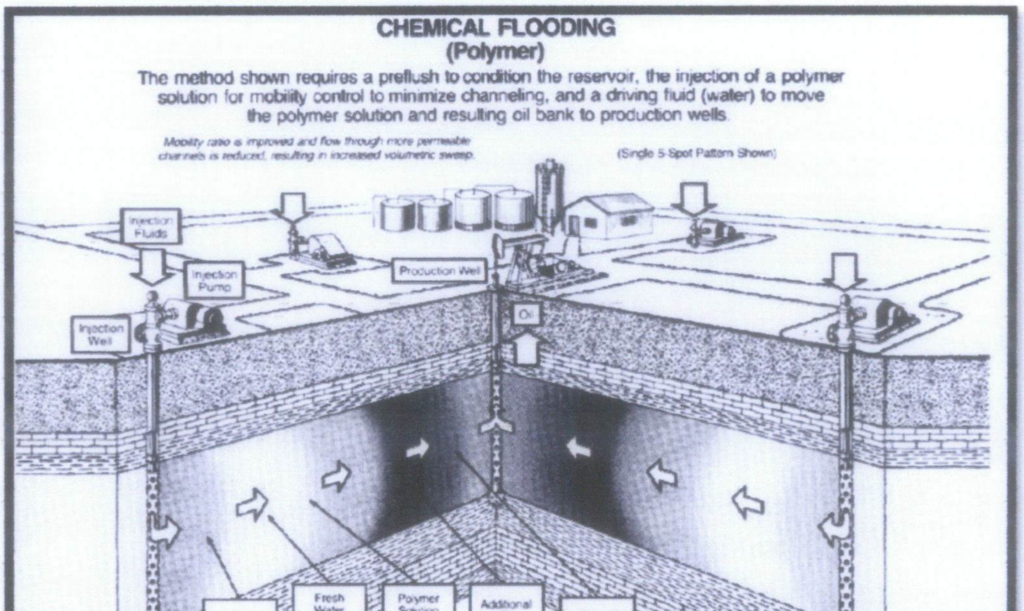


Figure 1: Polymer Flooding Process(Lindley, 2001)

Dilute partially hydrolyzed polyacrylamide (HPAM) acts as viscosifier in polymer flooding since it is widely used in the EOR field. The properties of the polymer solution are important since it is widely used in all applications especially in pipeline industry. In fluid flow operations such as multiphase crude oil transportation, drag reduction leads to an increased efficiency of operation during its transportation. Present study was carried out with a polymer additive (HPAM) as a drag reducing agent in oil and gas engineering (Sadiku-Agboola et al., 2011).

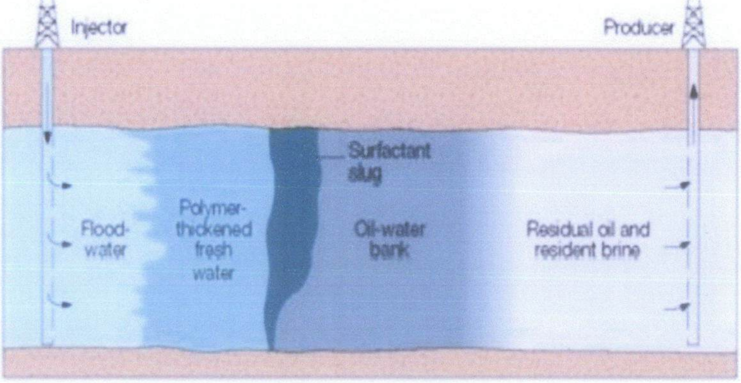


Figure 2: Enhanced Oil Recovery (*About Polyacrylamide*, 2012)

The science of rheology in polymer is concerned with the deformation and flow of matter and describes the interrelation between force, deformation and time. The term comes from Greek *rheos* meaning to flow (Adebowale, 2013). The measurement of rheological properties is applicable to all materials – from fluids such as diluted solutions of polymers and surfactants, to semi-solids such as pastes and creams, as well molten and solid polymers. Fluid rheology is used to describe the consistency of different products, normally by the two components that include viscosity and, which relates to the resistance to flow or its 'thickness'; and elasticity that is associated to its structure or 'stickiness'. Most rheological tests involve applying a force to a material and measuring its flow or change in shape (McClements, 2003). Many research have been done to understand the rheological behavior of HPAM since it is broadly used in pipeline and the field of oil and gas to enhance the viscosity of the transported liquid. In this research study, the rheological properties and behavior of HPAM is investigated further to see how it is affected on various conditions.

1.2 Problem Statement

Most of the studies carried out on rheological properties of HPAM that are aiming on the behavior in different temperatures prove that change at different temperatures affect the rheological properties. However, the studies that investigate on how modulus behavior reflects through viscoelasticity and the strength of HPAM are very scarce. In addition, effect of storage period may affect the viscoelasticity of HPAM solutions as well.

In order to know the improvement on HPAM behavior in EOR, this project paper was done to investigate on the **rheological properties** and **modulus behavior effects** subjected to:

- Master Concentrations of HPAM solution (ppm)
- Diluted Concentrations of HPAM solution (ppm)
- Effects of hydration period for both master and diluted concentrations

Rheological properties and modulus behavior effect subjected to its various concentration and time of hydration are expected to overcome this issue. The study on the polymer solution strength through the modulus behavior implies on how the strength of the polymer solution is affected through the implication of the shear and elastic moduli. The measurement of modulus behavior and rheological properties of any polymeric material is crucial in order to gain fundamental understanding of the process ability of that material.

1.3 Objectives & Scope of Study

Those parameters will be prepared to analyze the data on shear and elastic moduli profile. The main apparatus in this experiment is *Bohlin Gemini II* rheometer which offers complete rheological assessment with the flexibility to configure and optimize test conditions for all materials and application area. An experimental study will be conducted to investigate the rheological effect of shear and elastic moduli subjected to concentration and time of hydration difference of HPAM in a closed room temperature of 25°C. The test is done only at 25°C and not on the reservoir condition because the solvent trap located in the rheometer that can alter the temperature difference is not available during the project duration.

1.4 Relevancy of the Project

Polymer in oil and gas industry has become an essential element and many study and investigation have been done to maximize and varies the production of polymer material. Understanding the rheological properties enables us to optimize their process conditions, thus saving costs and potential waste. This also will introduce and describe more on the rheological theory of polymer fluids.

1.4.1 Feasibility of the Project

This project is involving project planning and experimental work stages. Research on work flow and preparation of the master concentration according to the difference in time of hydration at the a constant temperature will be done according to reliable sources such as previous findings, journals, articles, websites and related documents to prepare a good planning for the project. The theories and concept of the parameters involved in the experiment also will be emphasized in this study.

1.4.2 Significance of the Project

One key that makes polymer flooding is the most widely used chemical EOR techniques is its **viscoelasticity. Viscoelasticity** defined as a material that exhibit both viscous flow and elastic deformation. Viscoelasticity contribute towards improved displacement efficiency & oil recovery in polymer flood operations (Veerabhadrappa, 2012). However, the contribution of elasticity of viscoelastic polymers in various concentrations and the hydration period still remains largely unexplored. Hence, understanding the rheological properties and modulus behaviour is important to get the best out of viscoelastic polymers that can result in better oil recovery performance.

CHAPTER 2: LITERATURE REVIEW

2.1 Partially Hydrolyzed Polyacrylamide Solution

Partially Hydrolyzed Polyacrylamide (HPAM) solution is a linear copolymer of sodium acrylate and acrylamide monomer that was prepared by free radical polymerization. It contains amide groups (-$CONH_2$) and carboxylic groups (-COOH).This polymer is supplied in solid and liquid form. Since PAM is a highly water absorbent, there are certain periods of time needed for this polymer to be hydrolyzed to ensure the results is efficient on its applications. Polyacrylamide (PAM) and partially hydrolyzed polyacrylamide (HPAM) are synthetic, water-soluble polymers, showing unique shear-thickening properties.

HPAM solutions are broadly used as polymers for enhanced oil recovery, where the employment of this polymer as drag reducers becomes a current practice in the chemical industry. HPAM solutions are also used as polymers for water-based drilling mud that have proved effective and versatile for inhibiting troublesome shale formations(Kadaster et al., 1992). Another common use of polyacrylamide is in subsurface applications such as enhanced oil recovery, where high viscosity aqueous solutions can be injected to improve the economics of conventional water flooding (*About Polyacrylamide*, 2012).

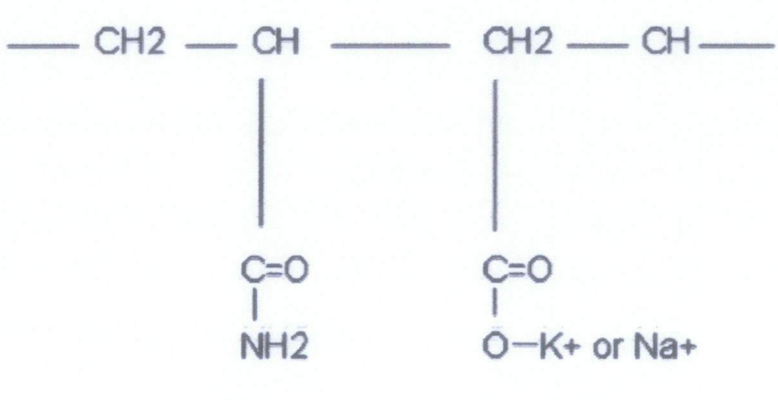


Figure 3: Structure of HPAM (Gao, 2013)

Ferreira and coworkers (Ferreira et al., 2006) made a research to find the potential major drag reduction mechanisms which evaluating the rheological behavior of HPAM (another commercial name of HPAM in oil and gas industry) and xanthan and diutan gums solutions that have drag reduction characteristics. It is expected that these polymers present a good drag reduction potential.

All these polymers are evaluated as drag reducers in a pressure drop flow loop. The final step is to correlate drag reduction tendency with rheological properties and molecular structure.

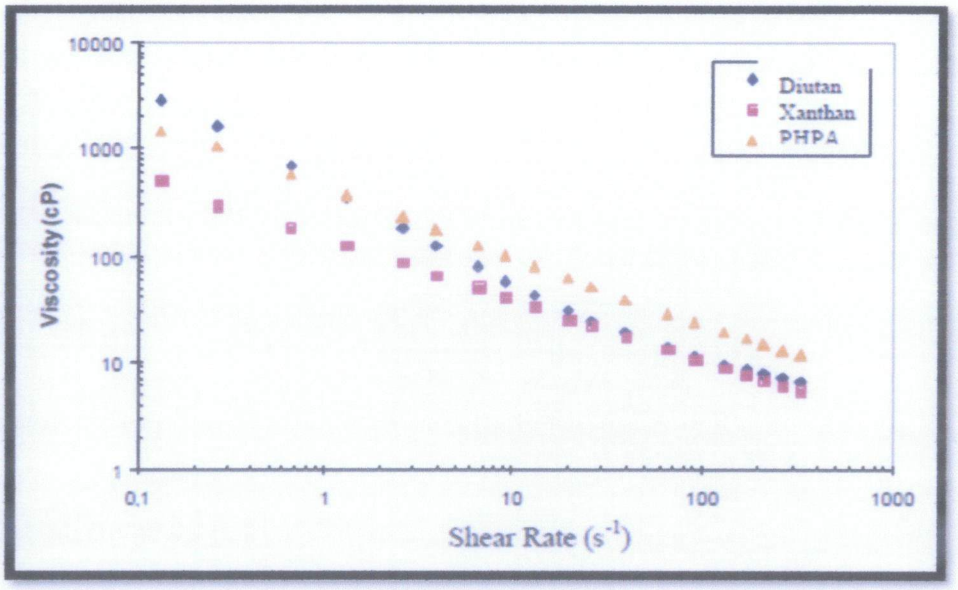


Figure 4: Viscosity curves for all polymers available (Ferreira et al., 2006).

Figure 4 shows the viscosity curves for the three polymers evaluated at same concentration and temperatures. It can be observed that, at low shear rates ($10s^{-1}$), the viscosity of HPAM is always higher than the diutan and xanthan gum. With the increasing of the shear rate, HPAM presents higher viscosifying capacity (Ferreira et al., 2006).That is the reason HPAM is broadly used in enhanced oil recovery. The proposed methodology proved to be adequate to evaluate rheology and drag reducing potential of polymeric solutions.

Another study of polymer flooding has been reported. Smith (Smith, 1970)which investigated the behavior of HPAM solutions in porous media on one class of HPAM that apparently is being tested widely as an additive for injection water. The investigation surveys some ways in which polymer solution properties are affected by polymer molecular weight, rock and fluid properties, flow rate and temperature. Results of laboratory tests show that the extent of polymer adsorption from solution may be quite high if the solution is very saline or is in contact with carbonate rock. They also suggested that solution salinity, flow rate, rock pore size and polymer molecular weight greatly influence the reduction of mobility and permeability by polymer solution (Ferreira et al., 2006).

In this study, an experimental work needed to be carried out to provide a comprehensive and wide-ranging study of rheological properties of HPAM solution. Polymer used in this study case is ZETAG 4120 (the commercial name of HPAM) which consists of 25% of polyacrylamide and 75% sodium acrylate. HPAM has been diluted with water to a concentration of 10, 20, and 50ppm within the temperatures at 25, 50, and 70°C. This various concentration and temperature measurement test were conducted to characterize the rheological behavior of HPAM in terms of elastic modulus and shear rate. In dilute HPAM solutions, factors like temperature and concentrations affect the shear and elastic moduli, shear stress and yield stress response, and shear thickening and shear thinning.

2.2 Viscosity& Rheology

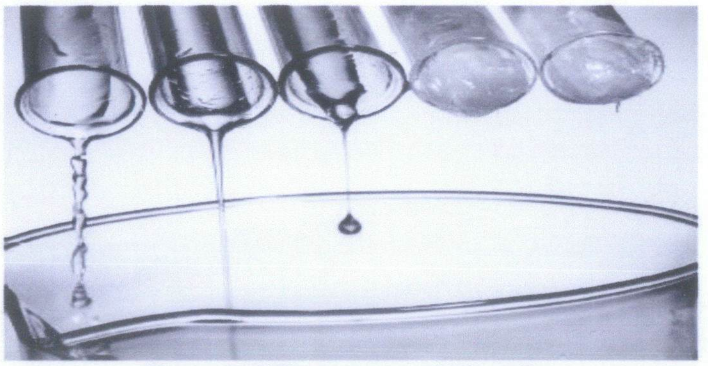


Figure 5: Different types of Viscosity

Viscosity is a measure of a fluid's resistance to flow. It describes the internal friction of a moving fluid. A fluid with large viscosity resists motion because its molecular makeup gives it a lot of internal friction.

Lewandowska (Lewandowska, 2007) describes her experimental results of rheological characteristics of polyacrylamide (PAM) and of partially hydrolyzed polyacrylamide (HPAM) (degree of hydrolysis up to 80%) in aqueous and aqueous/sodium chloride solutions with changing experimental conditions such as polymer concentration, temperature, solvent quality, and shear rate applied. She observed that the all-aqueous and aqueous/NaCl solution of PAM and of HPAM exhibited the non-Newtonian behavior with shear thinning and shear-thickening areas.

Many workers use the variation in rheological properties, namely solution viscosity to determine concentration. Another research about the viscosities of HPAM solution over a narrow temperature interval has been reported by Mungan (Mungan, 1968). He finds that the reduction in polymer solution viscosity is due simply to the reduction of the viscosity of water as the temperature raised.

Progress in both theoretical and experimental work on the viscosity characteristics of dilute polymer solutions has led us to the clarification of problems of the viscosities of concentrated polymer solutions, because the flow properties of these concentrated solutions involve many important problems. In developing these theories, Agboola et al. (Sadiku-Agboola et al., 2011) have attributed the enormous increase in the viscosity with increasing concentration to the formation of intermolecular linkages between polymers, and have adopted a working model of entangled polymer molecules.

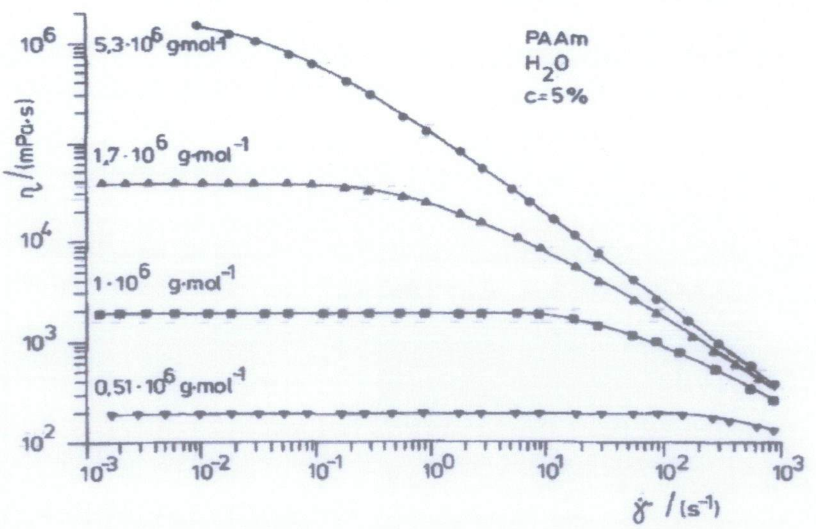


Figure 6: Viscosity as a function of shear rate for aqueous solutions of PAM of different molecular weights at a given concentration(Sadiku-Agboola et al., 2011).

The results from rheological investigations provide the mathematical description of the viscoelasticity behaviour of matter. Rheology analysis of polymers are including changes with temperature, concentrations, deformation rates, viscoelastic response as well as shear and elastic moduli in the analysis of all polymer types and formulations (Sadiku-Agboola et al., 2011).

A few researches also have been reported on the characterization of the elastic component of polymer solution. Yen et al., 2003 (Yen & Yang, 2003) studied the rheological behavior of polyacrylamide solution in the presence of various metal ions. The effect of temperature and types of metal ions on the rheological behavior of polyacrylamide-metal solution was systematically studied by using the power equation in the temperature range of 20-50°C and at polyacrylamide concentration of 0.5 wt%. The results indicated that the transition metal ions have a stabilizing effect on the viscoelasticity of PAM solution and the effect was proportional to the radius of the metal ion. It was found that greater shear stress of the polymer solution occurs in the presence of metal ions than with pure polyacrylamide solution (Yen & Yang, 2003). The authors' then compared the results of the rheological behavior of polyacrylamide–metal ion solution and the effect of temperature, as depicted in the figures below:

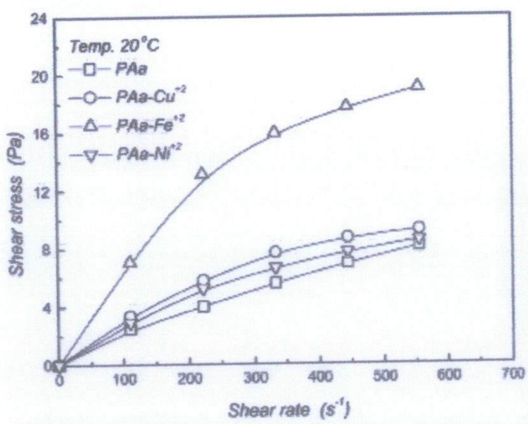


Figure 8: Dependence of shear stress on the shear rate for polyacrylamide solutions with different metal ions at 20 °C(Yen & Yang, 2003).

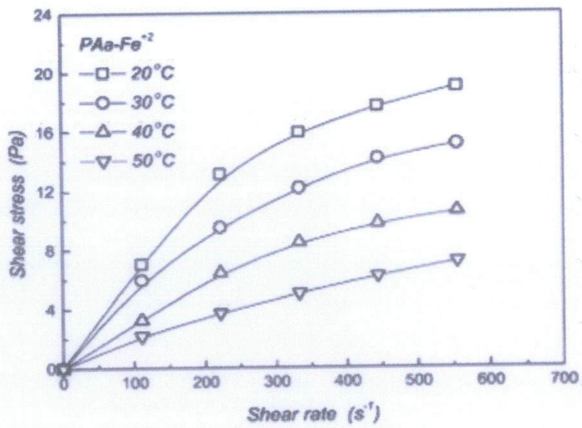


Figure 7: Dependence of shear stress on the shear rate for polyacrylamide–Fe+2 solution (Yen & Yang, 2003).

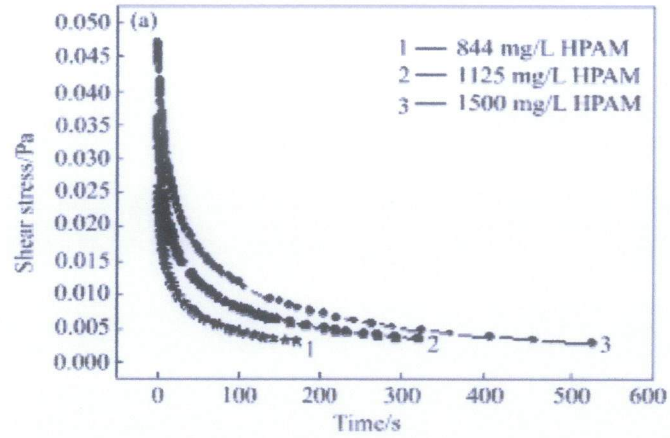
Fig. 7 shows the experimental results obtained for the polyacrylamide solutions at the concentration of 0.5 wt% containing three metal ions, indicating that the shear stress increases with increasing shear rate. The rheological behavior of polyacrylamide–iron ion solutions, which contained 25% mol ratio of iron ions to acrylamide units, was studied in the range 20–50 °C at0.5 wt% polyacrylamide concentration as shown in Fig. 8. It can be seen that at higher temperature, shear stress decreased and at higher shear rate, shear stress increased. Similar results were observed with other metal ions, for example Ni^{2+} and Cu^{2+}. From the experiments, it was found that the polyacrylamide–metal ion solutions studied in this paper behaved as pseudoplastic fluids at various temperatures.

2.3 Viscoelasticity

Viscoelasticity is a combination of viscosity and elasticity in varying amounts or mechanical characteristics exhibiting viscous flow and elastic deformation. Polymer solutions have very essential applications in the enhanced oil recovery and pipeline industry due to their unique viscoelastic properties related to shear modulus and elastic modulus. All materials have three categories of rheological behavior:

1) Viscous materials : all energy added is dissipated into heat
2) Elastic materials : all energy added is stored in the material
3) Viscoelastic materials : exhibit viscous and viscoelastic behavior

The phenomenon of long-term viscosity decrease of aqueous polyacrylamide solutions has been investigated with regards to its molecular origin. Zhao Feng et al. describe the characterization of viscoelastic properties of hydrolyzed polyacrylamide using the stress recovery experiment. In this study, the viscoelastic properties of partially hydrolyzed polyacrylamide (HPAM) solution have been investigated. Fig. 9 is plots of the shear stress recovery curves for HPAM solution at various concentrations(Zhao et al., 2004).



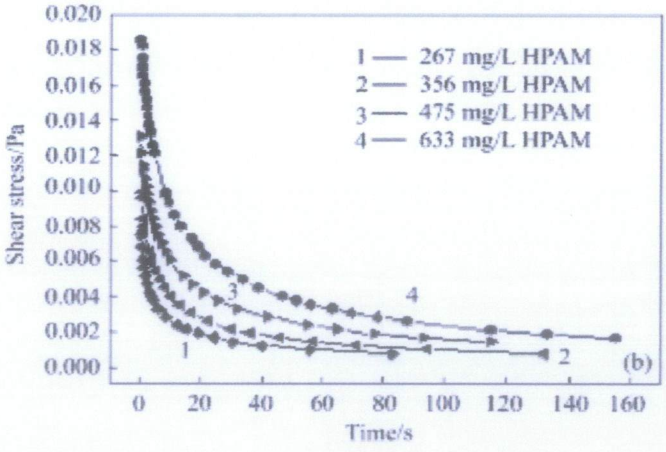


Figure 9: Plots of stress recovery curve for HPAM solution at various concentrations(Zhao et al., 2004).

Results show that the viscoelastic properties of HPAM increase with HPAM concentration increasing (Zhao et al., 2004). The characterization of the viscoelastic properties of HPAM solution using the shear stress recovery experiment is very convenient. The concentration show better results on the strength of viscoelastic properties with the increase of HPAM concentration.

2.4 Shear and Elastic Modulus

Normal stresses will appear when shearing a viscoelastic fluid and can result in different flow behavior from Newtonian fluids. For design of products, rheometric measurements are made to create the properties of elastic material such as yield value and gel strength. When oscillatory shear measurements are performed in the linear viscoelastic regime, the storage modulus G' (elastic response of HPAM) and loss modulus G" (viscous behavior of HPAM) are independent of the strain amplitude ("Rheology," 1998).

Shear modulus or modulus of rigidity is a ratio coefficient of elasticity for a shearing force. It can be describes as:

$$\text{Shear Modulus}, G = \frac{ShearingStress}{ShearingStrain}$$

The unit of shear modulus is normally used as Newton per meter square Nm^{-2} and Pascal (Pa).

2.4.1 Storage (Elastic) and Loss (Viscous) Modulus

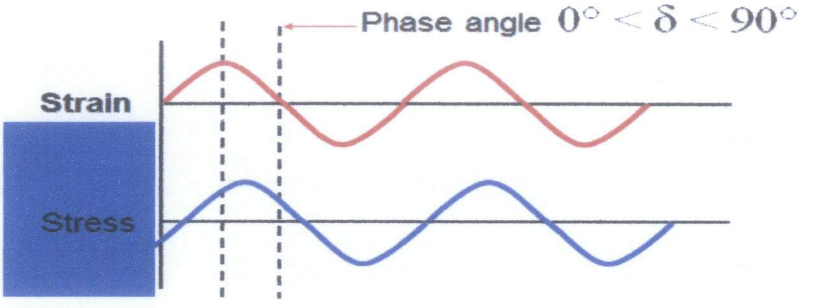


Figure 10: Strain Sweep

A large value of G' determines elastic gel properties of the polymer being analyzed. A phase angle, δ of 0^0 means a perfectly elastic material (storage modulus) and at 90^0 means a perfectly viscous material (loss modulus). Frequency gives some information about the gel strength as (Alcantara, 2013):

1) G' large slope curve determines the low strength and
2) G' small slope curve determines the high strength

The study of effects of polymer elasticity on enhanced oil recovery by core flooding and visualization experiments were carried out by (Veerabhadrappa, 2012) is very significant in investigating the shear and elastic modulus as well as loss and storage modulus of HPAM(Khan et al., 2009)(Khan et al., 2009) (Khan et al., 2009). Radial core experiments were conducted with seven HPAM samples having different average molecular weight. In this study, he conducted a frequency tests on HPAM samples at a range of frequency from 0.01 Hz to 1 Hz to observe the viscous modulus and elastic modulus behaviour against angular frequency.

HPAM Sample	Mass Fraction of HPAM Grades				Avg. Molecular Weight ($M_{w,B}$)
	3630	3330	3130	AB005	
HPAM 1	0	0	1.0	0	2.000E+06
HPAM 2	0.11	0.15	0.41	0.33	2.008E+06
HPAM 3	0.25	0.00	0.35	0.40	2.043E+06
HPAM 4	0.20	0.16	0.15	0.49	2.006E+06
HPAM 5	0	1.0	0	0	8.000E+06
HPAM 6	0.45	0.25	0.30	0	8.000E+06
HPAM 7	0.59	0.19	0.05	0.17	8.000E+06

Table 1: Composition and weight average molecular weights (AMW) of HPAM samples(Veerabhadrappa, 2012).

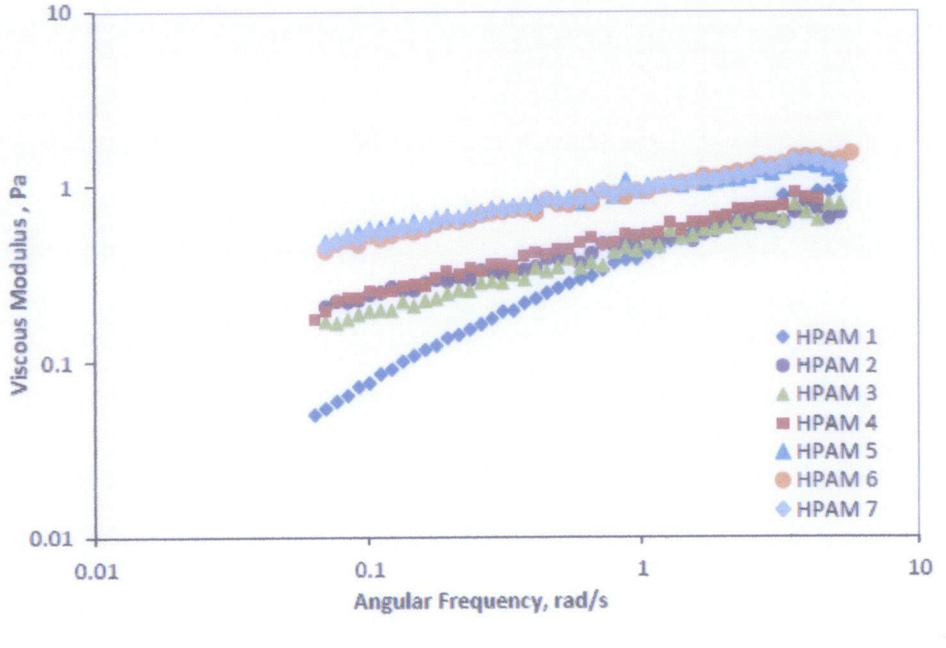


Figure 11: Viscous Modulus vs. Angular Frequency for HPAM-1 to HPAM-7(Veerabhadrappa, 2012).

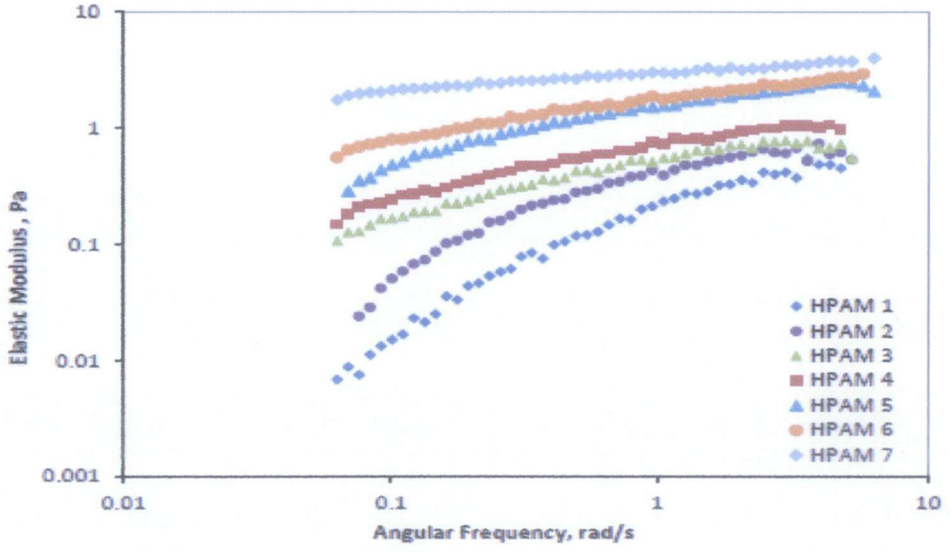


Figure 12: Elastic Modulus vs. Angular Frequency for HPAM-1 to HPAM-7 (Veerabhadrappa, 2012).

Viscous Modulus vs. Angular Frequency	Elastic Modulus vs. Angular Frequency
• HPAM-5 to 7 with higher AMW are greater than HPAM-1 to 4 with low AMW. • HPAM with higher AMW will have higher viscous modulus .	• Even same AMW, HPAM-7 has the highest elasticity (Veerabhadrappa,2012).

Table 2: Results of Viscous Modulus and Elastic Modulus vs. Angular Frequency

The cumulative and breakthrough oil recovery analysis provided a good measure of polymer screening based on average molecular weights of polymers. Results from the radial core flooding experiments show that higher recovery can be achieved with polymers having higher elasticity.

The frequency sweep test measures viscoelastic properties of tested materials as a function of frequency has been done by Shaw and MacKnight (Shaw & MacKnight, 2005). During the test, a varying frequency is applied on tested samples with a constant value of stress. The test consists of the delay and sampling intervals: frequency is applied during the delay time and the phase shift δ between the stress and the strain as well as the complex modulus G* is measured. The complex modulus is given by:

$$G^{*} = G' + iG" = \frac{\gamma_m}{\sigma_m}$$

The elastic modulus G' is usually referred to as the storage modulus to describe the elastic storage of energy, because strain is recoverable in elastic materials. The viscous modulus $G"$ is referred to as loss modulus to describe the viscous dissipation or loss of energy due to permanent deformation in flow.

The parameter G' and $G"$ (Shaw & MacKnight, 2005) are given as follows:

$$G' = G^{*} \cos\delta = \frac{\gamma_m}{\sigma_m}\cos\delta$$

$$G' = G^{*} \sin\delta = \frac{\gamma_m}{\sigma_m}\sin\delta$$

Where;

γ_m = amplitude of applied strain

σ_m = stress related nearly to strain

2.4.2 Oscillation Tests

Oscillation tests provide important information related to the viscoelasticity of polymer solutions. Through these tests, it gives a clear indication of the behaviour of the sample which can determine whether viscous nature or the elastic nature of polymer is dominating over a given range of shear or angular frequency (Shaw & MacKnight, 2005).

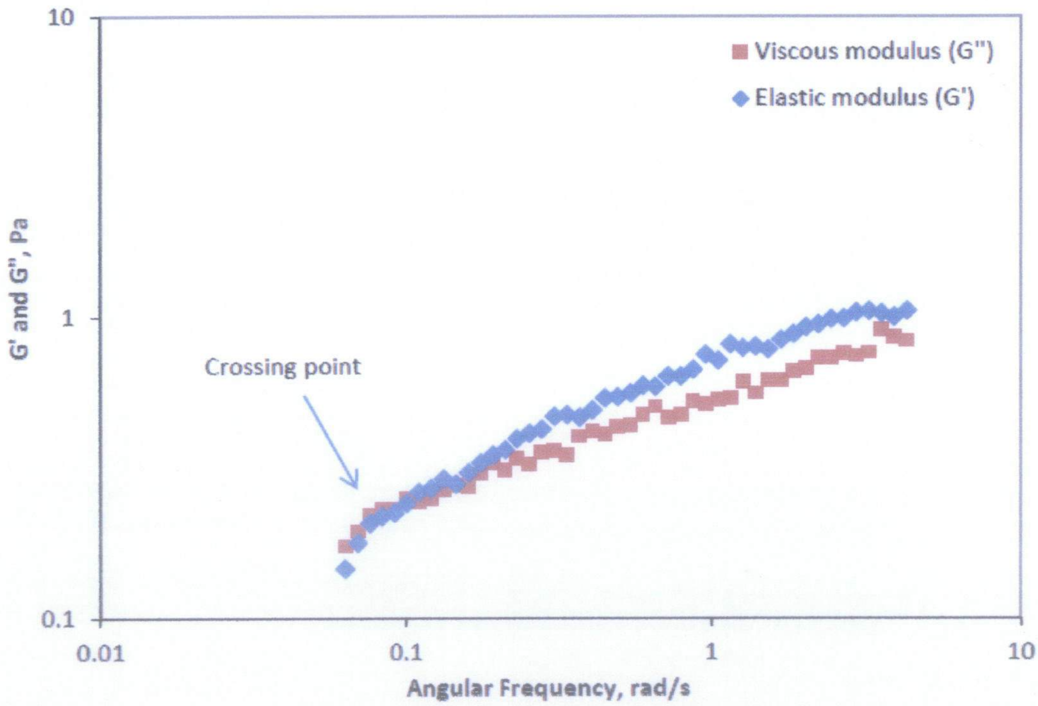


Figure 13: Graph of G' and G" (Pa) vs Angular Frequency (rad/s)(Veerabhadrappa, 2012).

Figure 13 shows the viscous modulus and elastic modulus of HPAM-4 and the cross-over point (Veerabhadrappa, 2012). From the literature (Meng et al., 2008), the G' and G'' increase with polymer concentration and viscoelasticity becomes more prominent. A cross-over point present in the graph plotted where the corresponding frequency is called the specified frequency (SF). The SF moves to lower frequency as the concentration of HPAM increases when the angular frequency is less than SF, thus G'' is greater than G', i.e. viscous effect is more dominate. When the angular frequency is more than SF, G' is greater than G'' which implies that the elastic effect is more dominating.

From all the research, the applications of HPAM throughout various industries are broadly used especially in investigating the rheological behavior of HPAM i.e. viscosity, viscoelasticity, and shear and elastic modulus. However, the viscoelasticity of HPAM is affected by the concentrations of master and dilute solution and different temperatures is not yet being investigated very specifically. To clarify on the stated matter, further research and investigation on rheological properties of HPAM is important to ensure the best decision has been made.

Table 3 summarizes the previous work done by the researchers and the analysis towards the results and methodologies used relative to this project.

Table 3: Summary of previous work done on investigating the viscoelasticity and rheological behavior of the HPAM solutions.

No	Author	Title	Objective	Method	Result
1	Veerabhadrappa, 2012 (Veerabhadrappa, 2012)	Study of Effects of Polymer Elasticity on Enhanced Oil Recovery by Core Flooding and Visualization Experiments	To explain and examine **the effect of elasticity** of seven samples of HPAM with different **Average Molecular Weight (AMW)** towards improved oil recovery	All these polymers (HPAM, Xanthan &Diutan gums) will be evaluated as drag reducers in a pressure drop flow loop in terms of: 1) Constructing flow loop to quantify the frictional losses 2) Structural & Composition Characterization 3) Rheological Characterization 4) Thermal Stability 5) Flow loop test	Focusing only on the Rheological Characterization graph - Polymer with higher elasticity gives higher recovery. HPAM samples with **higher AMW will have higher shear viscosity and elasticity.**
2	Zhao F. et al, 2004 (Zhao et al., 2004)	Characterization of viscoelastic properties of hydrolyzed polyacrylamide using the stress recovery experiment	To study **the effect of HPAM concentration on the viscoelastic properties** of HPAM solution by two recovery time using the stress recovery experiment.	The stress creep/recovery experiment was performed using a cone-and-plate system. The viscoelastic behavior of HPAM solution consists of fast recovery process which corresponding to the local recovery movement of HPAM chain segments. The other is a slow recovery process corresponding to the reorientation and recovery movement of HPAM chain coils when the polymer molecular coils are forced to deform due to the shear stress input.	The viscoelastic properties of HPAM increase and it becomes stronger with the increase of HPAM concentration.

3	Hou J. et al, 2005 (Hou et al., 2005)	The role of viscoelasticity of alkali/surfactant/polymer solutions in enhanced oil recovery	To treat alkali scale related problems encountered in the alkali/surfactant/polymer (ASP) flooding pilot area of Daqing Oilfield.	The effect of alkalis and surfactants on the viscoelasticity of ASP solutions are studied using HAAK RS-150H type rheometer (made in Germany). The polymer used in the experiments is partially hydrolyzed polyacrylamide (HPAM).	The studies indicate that alkali has adverse effect on the **viscoelasticity** of an ASP solution. A high concentration of alkali can cause the HPAM molecular chains curl up and can form alkali scales. The viscoelasticity of an ASP solution contributes more to the oil recovery of a heterogeneous reservoir. **The stronger the heterogeneity is, the greater the effect of viscoelasticity on the oil recovery.**
4	Jung J. C. et al, 2012	Rheology and Polymer Flooding Characteristics of Partially Hydrolyzed Polyacrylamide for Enhanced Heavy Oil Recovery	Polymer flooding characteristics of partially hydrolyzed polyacrylamide (HPAM) solution with the addition of NaOH were examined in homogeneous glass-bead packs. The heavy oil recovery in unconsolidated sandstone formations by applying the alkali-polymer flooding was observed.		

CHAPTER 3: METHODOLOGY

3.1 Project Activities

This chapter will discuss the detail explanation of methodology that is used upon completing the project. A master solution is a concentrated solution that will be diluted to a lower concentration for definite use. In this project, both master solution and diluted solution will be used to measure the viscoelasticity of HPAM in different time of hydration. A cone-plate method of rotational viscometry is used in this experiment to analyze the viscoelasticity of HPAM respectively. The experimental procedure of the preparation method is as follows:

Research on Rheology of Partially Hydrolyzed Polyacrylamide (HPAM)

↓

Preparation on Master solution concentration of HPAM

↓

Prepare Diluted solution concentration from master solution

↓

Setting constant temperature of rheometer at 25°C

↓

Repeat analysis of data with different time of hydration

↓

Gathering the data

↓

Analyzing data

↓

Conclusion

3.2 HPAM Master Solution Preparation

Three master solution concentrations at 700ppm, 500ppm, and 300ppm of HPAM are to be prepared for this experiment. The method of mixing is being applied:

a) ZETAG 4120 powder (the commercial name of HPAM) is sprinkled evenly on a flat bottom tank.

b) 10 liters of DI (deionized) water is poured inside the tank at once, allowing the polymer to be dispersed around the tank.

c) The sprinkle powder and DI water will be mixed and then allowing the solution to be placed for 24 hours at room temperature.

d) Figure 14 illustrates the preparation of master solution method.

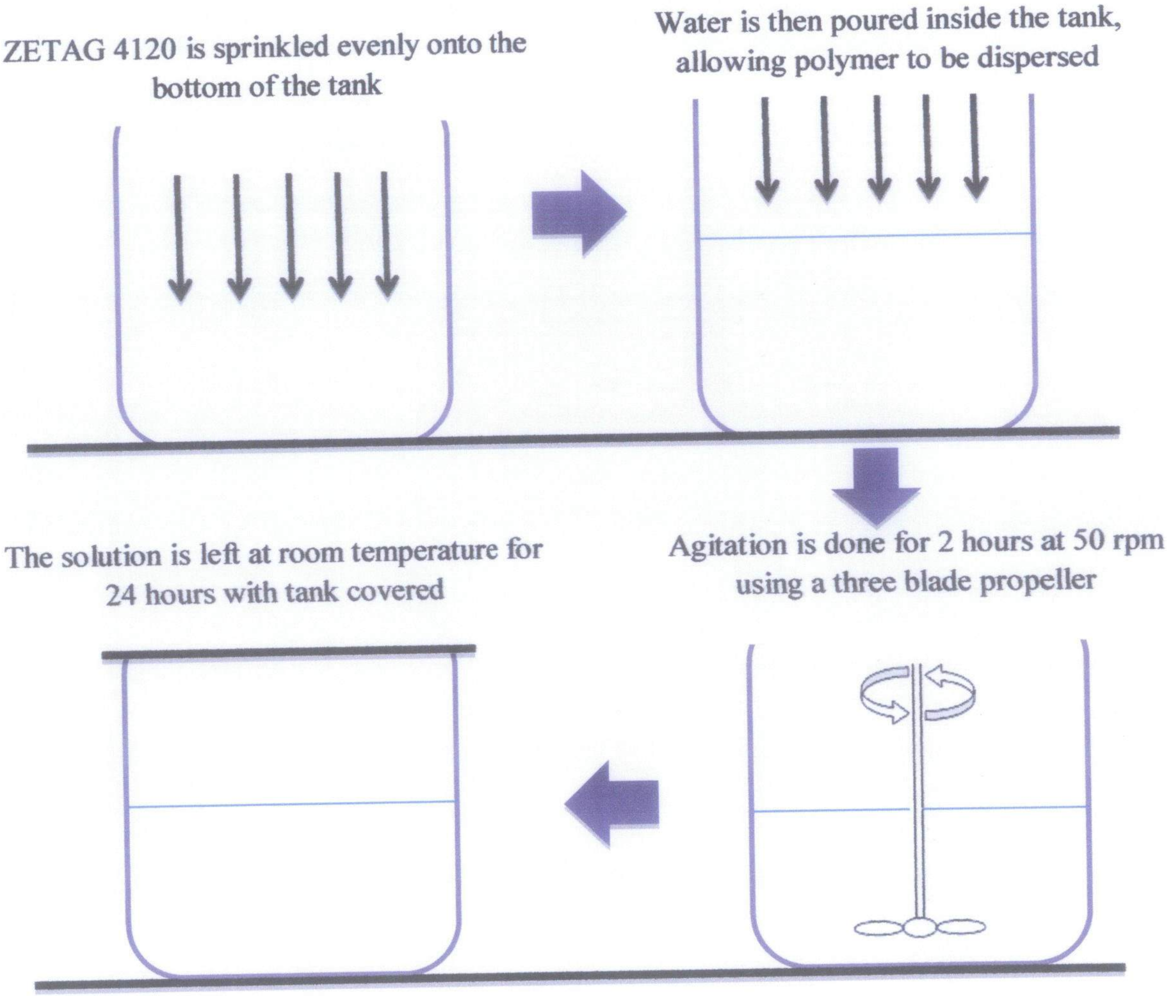


Figure 14: Polymer Master Solution Preparation

Dispersing polymer powder at bottom of the tank and pouring water above it will produce a uniformly-distributed polymer viscosity. Both components of powder and water can be mixed properly and uniformly during process of agitation using three blade propeller.

The solution is stirred for 2 hours at a low speed of 50 rpm constantly. The high speed of stirring can avoid high shear rate that can cause the solution to degrade or damage through scissoring of polymer chains. The solution is then placed in the conical flask, covered and left for a day, a week and a month to allow the process of hydration on the solution and to avoid any impurities contaminating the solution.

3.3 Dilution from Master Solution

After 24 hours process of hydration, the master solutions will be diluted to 10ppm, 20ppm, and 50ppm solutions respectively using DI water. Dilution calculations mainly involve figuring out the final concentration or volume (depending on what's given and what's known) after a volume or concentration has been changed.

Dilution process is done to stimulate the exact concentration injected to the pipeline during drag reduction process and dilution equation follows the relation given below:

$$C_m V_m = C_d V_d$$

Where,

C_m = concentration of master solution (ppm)

C_d = concentration of dilute solution (ppm)

V_m = volume of master solution required for dilution (L)

V_d = volume of dilute solution (L)

Table 4: Amount of Dilution Concentrations from Master Solutions

Master Concentration (ppm)	Dilute Concentration (ppm)
700	10, 20, 50
500	10, 20, 50
300	10, 20, 50

After preparing diluted solution for three respective concentrations for each master solution, each of the dilution solution will be tested using *Bohlin Gemini 2* rheometer to identify the viscoelacticity characteristics as well as their respective rheological properties and behavior.

3.4 Laboratory Testing & Measurement

A rotating rheometer introduced in this experiment will be *Bohlin Gemini 2* rheometer as shown in Figure 15 that will measure, test, and analyze data on the viscoelasticity and rheological properties for both master solution and diluted solutions. This rheometer is optimized for both strain controlled and stress controlled operation.

Table 5 represent four modes that operating in *Bohlin Gemini 2* rheometer shown below:

Table 5: Operating modes of Bohlin Gemini Rheometer 2 ("Bohlin Gemini ", 2009)

Range of operating modes	Description
Oscillation test	Measures the dynamic viscous, G" and elastic, G' properties and phase angle as a function of frequency in Hertz (Hz)
Viscometry	Measures viscosity as a function of shear stress, σ or shear rate, $\dot{\gamma}$
Relaxation	Measures relaxation modulus (step strain experiment changes) as a function of time in second (s)
Creep and Creep Recovery	Measures the creep compliance and compliance resurgence as a function of time in second (s)

In order to evaluate these polymer solutions of 300, 500, and 700ppm as well as their respective dilution in elastic and viscous modulus, the rheometer is set as Oscillation Test. It is conducted at Biohydrogen Lab at block P, Universiti Teknologi Petronas.

Figure 15: Bohlin Gemini 2 II rotational rheometer

By conducting the rheometer test, temperature of 25°C will be selected to determine the results of the viscoelastic modulus behavior of the master and diluted solution.

3.5 Key Milestone & Gantt Chart

FYP 1 Milestone

NO	ACTIVITY	MAY		JUNE				JULY					AUGUST			
		W1	W2	W3	W4	W5	W6	W7		W8	W9	W10	W11	W12	W13	W14
1	Selection of Project Topic								MID-SEMESTER BREAK					AIDILFITRI		
2	Preliminary Research Work on the Objective & Scope of Study															
3	Research on History of Polymer Application in Industry															
4	Research on Rheological Behavior of HPAM															
5	Study on the Viscosity, Viscoelasticity of HPAM															
6	Study on HPAM Preparation															
7	Submission of Extended Proposal Defence															
8	Proposal Defence															
9	Preparation of Interim Report (Review on the Effect of Temperature and Concentration of Polymer Solution)															
10	Draft Interim Report															
11	Submission of Interim Draft Report															
12	Submission of Interim Report															

Process

Suggested Milestones

FYP II Milestone

NO	ACTIVITY	SEPT		OCT				NOV				DEC				JAN
		W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14	W15
1	Preparing Master and Dilute solution for First batch of HPAM solutions	■	■	■	■	■	MID-SEMESTER BREAK	■	■	■						
2	Submission of Progress Report								●							
3	Preparing Master and Dilute solution for Second batch of HPAM solutions			■	■	■		■	■	■	■					
4	Analyse results of Oscillation tests	■	■	■	■	■		■	■	■	■					
5	Pre-SEDEX											●				
6	Final Report and Dissertation Draft									■	■	■				
7	Submission of Draft Report												●			
8	Submission of the Dissertation (Soft bound)													●		
9	Submission of the Technical Paper													●		
10	Oral Presentation/Viva														●	
11	Submission of the Dissertation (Hard bound)															●

■ Process

● Suggested Milestone

CHAPTER 4: RESULTS AND DISCUSSION

The rheometer software that measured using the cone and plate geometry was set as the 'oscillation test'. The plates used are ETC cone 5.4°/40mm for more viscous (master) solution and 2.5°/25mm for less viscous (dilute) solution. The results between master and dilute concentrations as well as the time of hydration were compared to study the rheological properties of HPAM. The suggested hydration times are one day, one week, and one month.

4.1 Comparison between master solutions at one day hydration

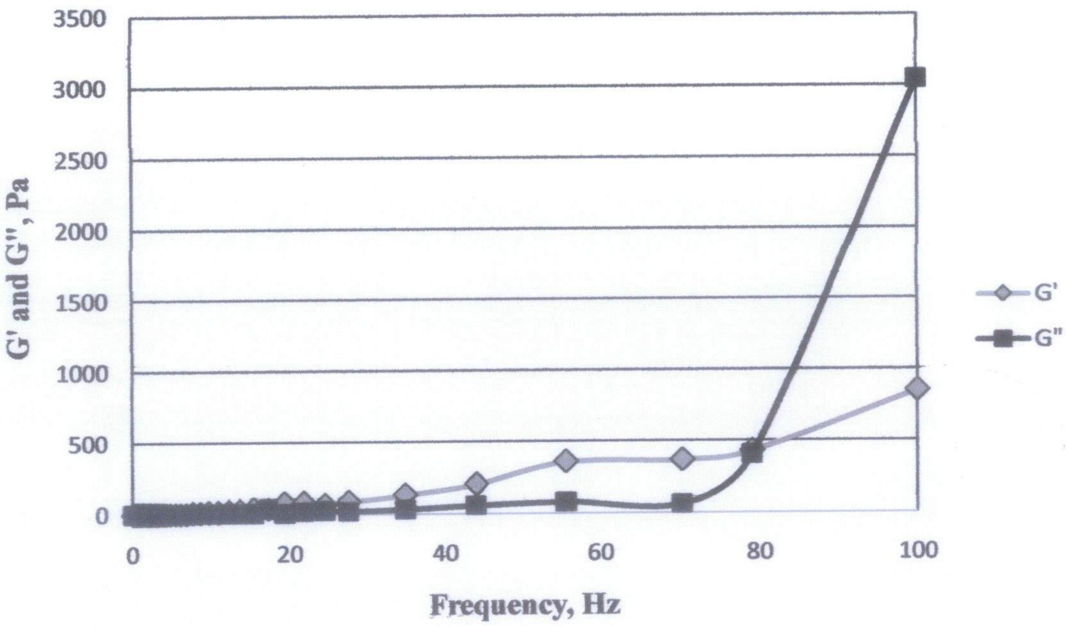


Figure 16: G' and G" profile for HPAM solutions for 700ppm at one day hydration

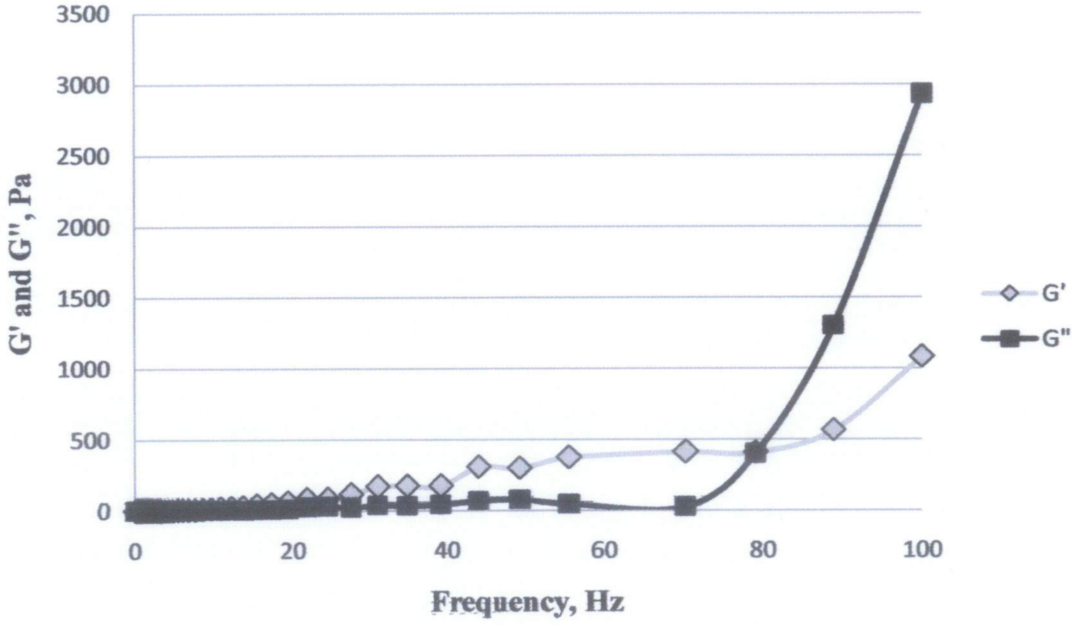


Figure 17: G' and G" profile of HPAM solutions for 500ppm at one day hydration

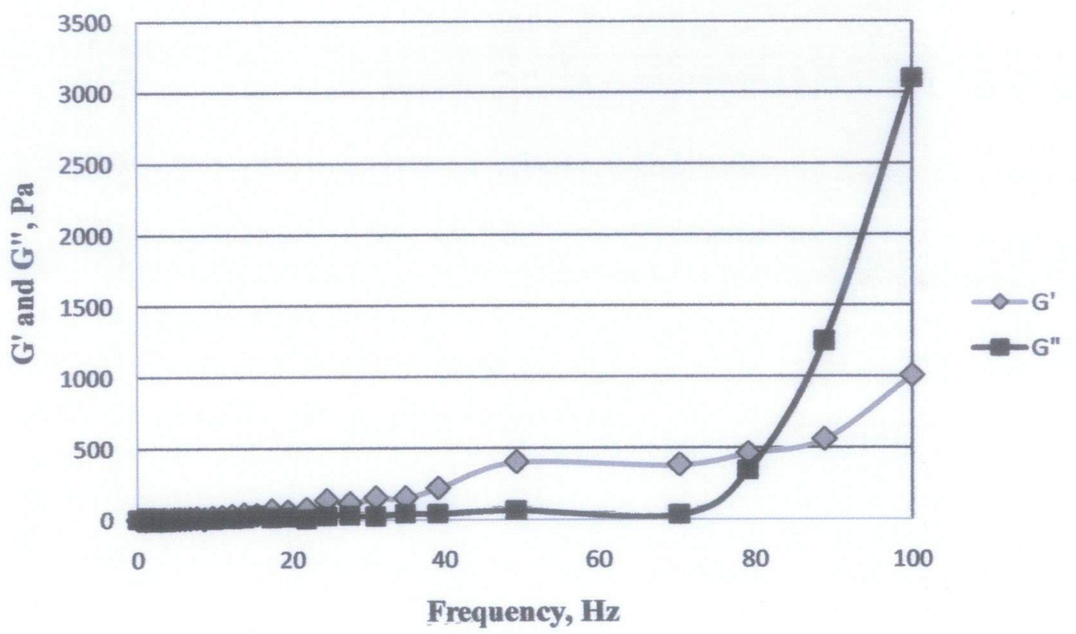


Figure 18: G' and G" profile of HPAM solutions for 300ppm at one day hydration

The results obtained from Figure 16, Figure 17, and Figure 18 shown is the rheological properties of the master solution of 700ppm, 500ppm and 300ppm at constant temperature of 25°C. All three HPAM solutions exhibit elastic and viscous modulus behavior. Within one day of hydration, the concentration affects the value of G' and G" (Pa).

Figure 16, Figure 17, and Figure 18 represent each of the modulus profile of 700ppm, 500ppm and 300ppm proved the higher value of G" than G'. In terms of viscoelastic properties, this polymer solution represent towards viscous rather than elastic properties.

Meanwhile, the master solution of 700ppm and 300ppm had higher value of G" which is more than 3 kPa compared to 500ppm which the value of G" is below 3 kPa. The intersection of the viscous, G" and elastic modulus, G' in the graphs signifies the **relaxation time** occurs at the moment. Relaxation time is the time constant of a curve system to return to equilibrium after an interference with the polymer chains. From this experiment, it happens in between approximately 79.12 to 82.15 Hz within 12 minutes of oscillation test. During that time, the polymer solutions behave more likely to viscoelastic properties.

For all solutions of 700ppm, 500ppm, and 300ppm, the viscous characteristic was more prominent compared to elastic recovery during flow. The polymer solutions were having high viscosity and consequently these solutions need a very huge amount of stress to cause the strain to be in a small amount.

4.2 Comparison of the Elastic Modulus, G' and Viscous Modulus, G" of the master solutions at one day hydration

4.2.1 Elastic Modulus, G' of master solutions

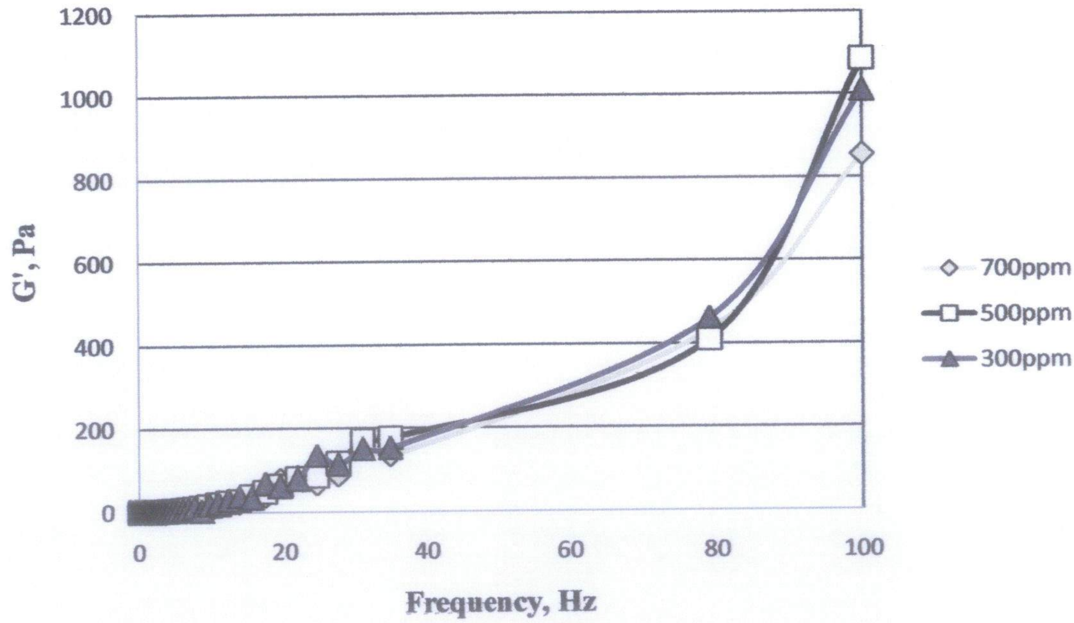


Figure 19: Elastic Modulus, G' profile of HPAM master solutions at one day hydration

Figure 19 elaborates more on the elastic behavior of three master solutions. The G' value for 700ppm, 500ppm and 300ppm HPAM solutions had rapid increasing at approximately 79.12 Hz, where the viscoelasticity deformation occurs at this point throughout time. For 500ppm, the value of G' was quite high to be compared with other master solutions and this shows that the 500ppm exhibit towards elastic behavior than the other two master solutions. Meanwhile, the value of G' for 700ppm solution behaves less elastic than 300ppm and 500ppm.

4.2.2 Viscous Modulus, G" of master solutions

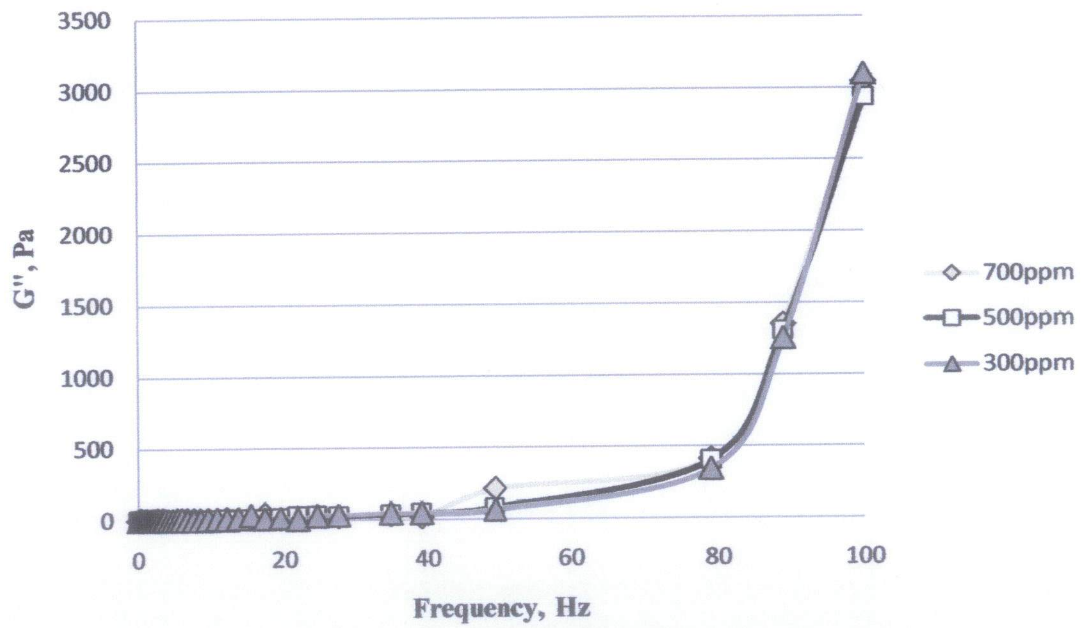


Figure 20: Viscous Modulus, G" profile of HPAM master solutions at one day hydration

Based on Figure 20, the graph plotted demonstrate more on the viscous behavior of three master solutions, which is the G" value for 700ppm, 500ppm and 300ppm HPAM solutions also increased at approximately 50 Hz, same as in Figure 19, where the deflection occured at this point throughout time. This time G' value of 300ppm was higher to be compared with other master solutions and this shows that the elastic behavior of 300ppm was significant than the other two master solutions. The viscous behavior was prominent for both of 700ppm and 500ppm solutions. However, from the data given from rheometer, the value of the solution of 700ppm at 100 Hz shows higher value of G" followed by 500ppm.

Frequency, Hz	Viscous Modulus, G" (Pa)		
	700ppm	500ppm	300ppm
100	3041	2929	3096

4.3 Comparison between diluted solutions from 700ppm at various time hydration

4.3.1 One day hydration period for 10, 20, and 50ppm dilutions

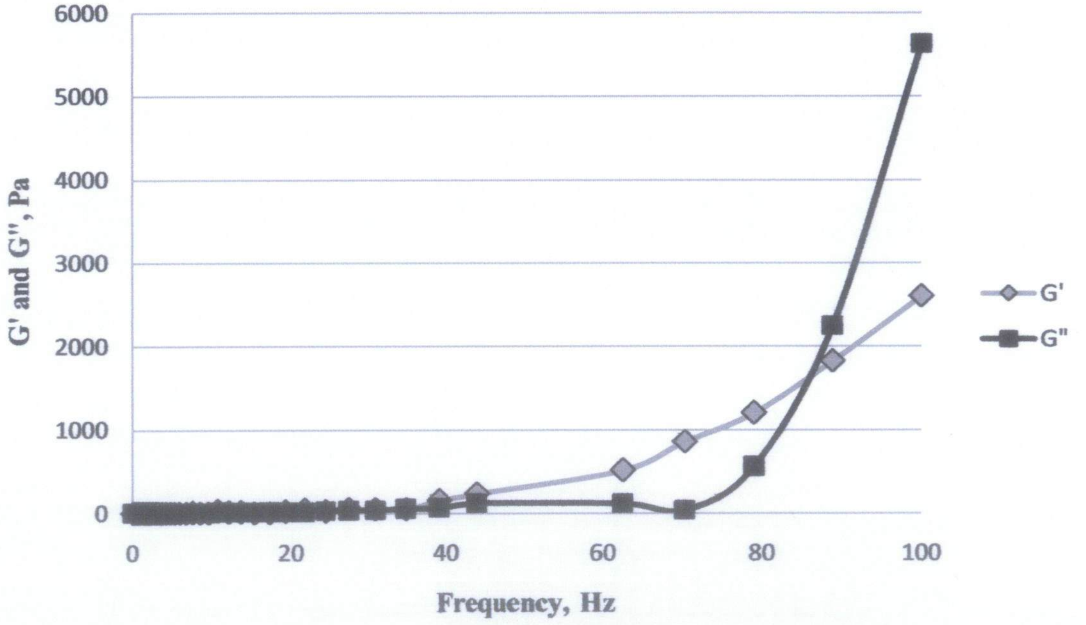


Figure 21: *G' and G" profile of HPAM diluted solutions of 10ppm from 700ppm master solution*

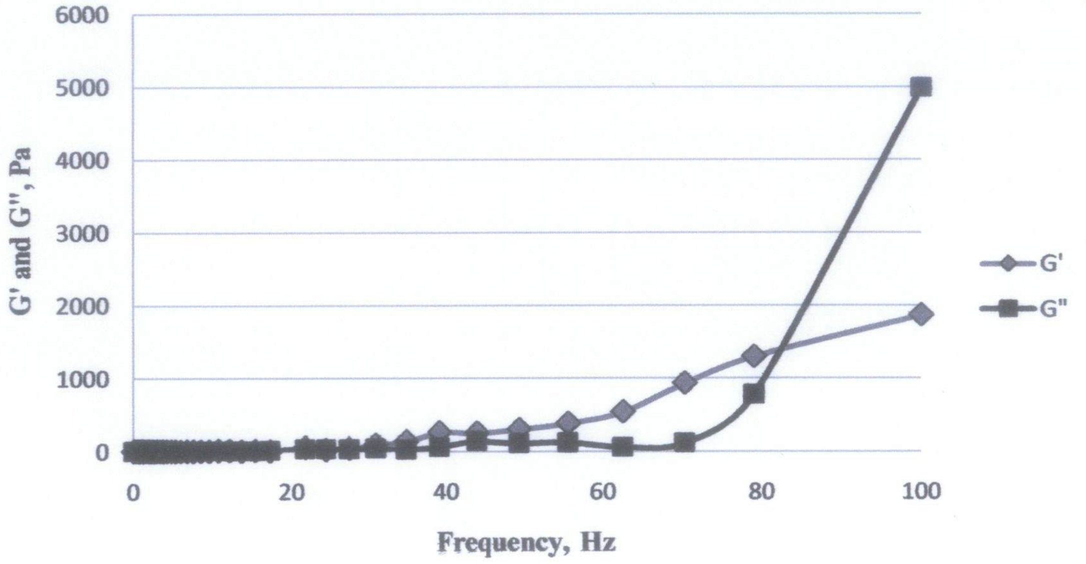


Figure 22: *G' and G" profile of HPAM diluted solutions of 20ppm from 700ppm master solution*

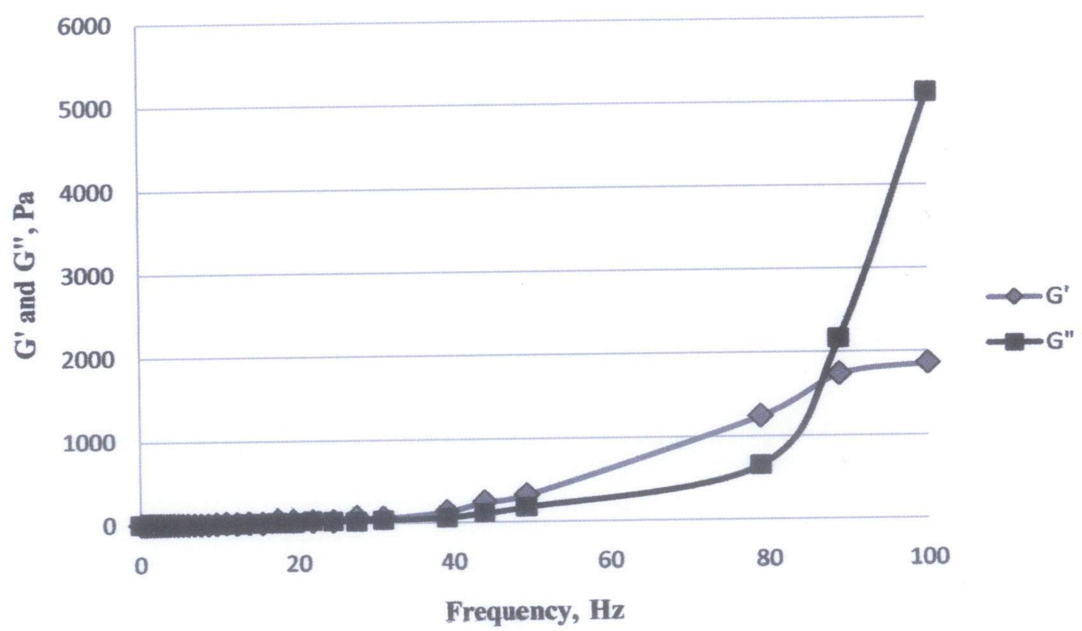


Figure 23: G' and G" profile of HPAM diluted solutions of 50ppm from 700ppm master solution

From **Figure 21**, **Figure 22**, and **Figure 23** shown, the diluted solution 10ppm of 700ppm, 500ppm, and 300ppm had higher G'' value than G' after one day left for hydration. The graph plotted can be concluded that all diluted solutions from 700ppm represent more viscous properties than elastic properties. In comparison between all the diluted solutions in terms of viscous behavior, 10ppm solution behaves more towards viscous properties followed by 50ppm solution.

4.3.2 One week hydration period for 10, 20, and 50ppm dilutions

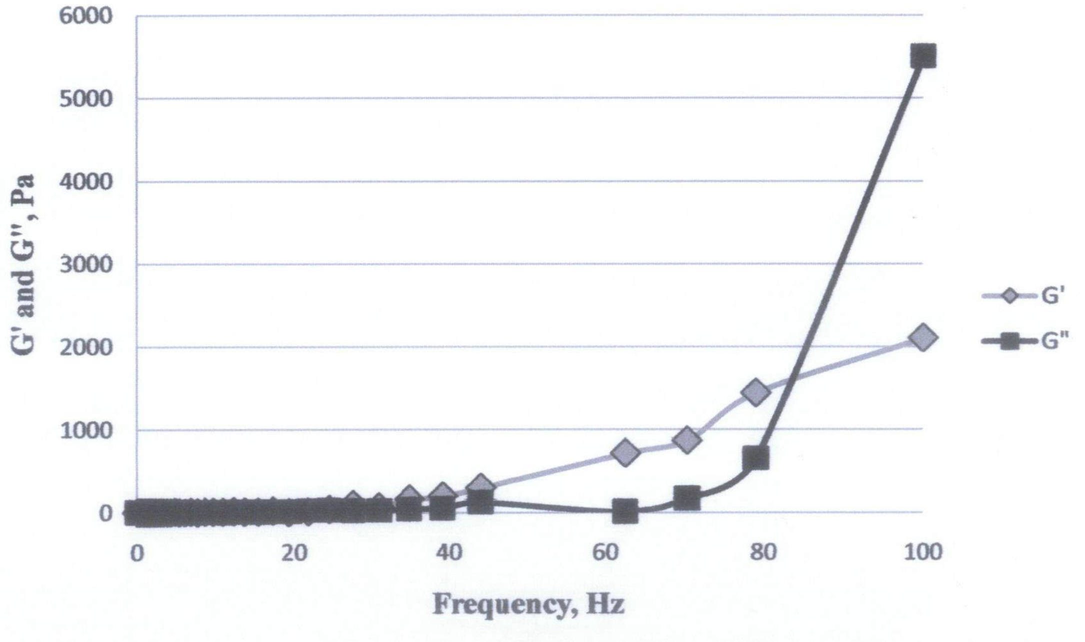


Figure 24: G' and G" profile of HPAM diluted solutions of 10ppm from 700ppm master solution

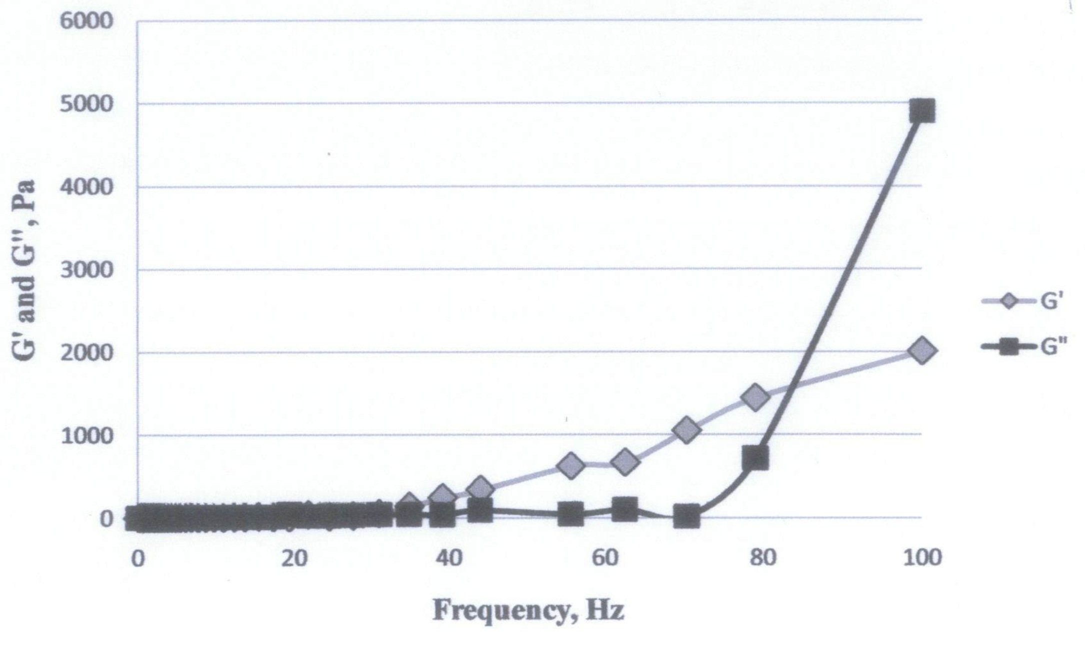


Figure 25: G' and G" profile of HPAM diluted solutions of 20ppm from 700ppm master solution

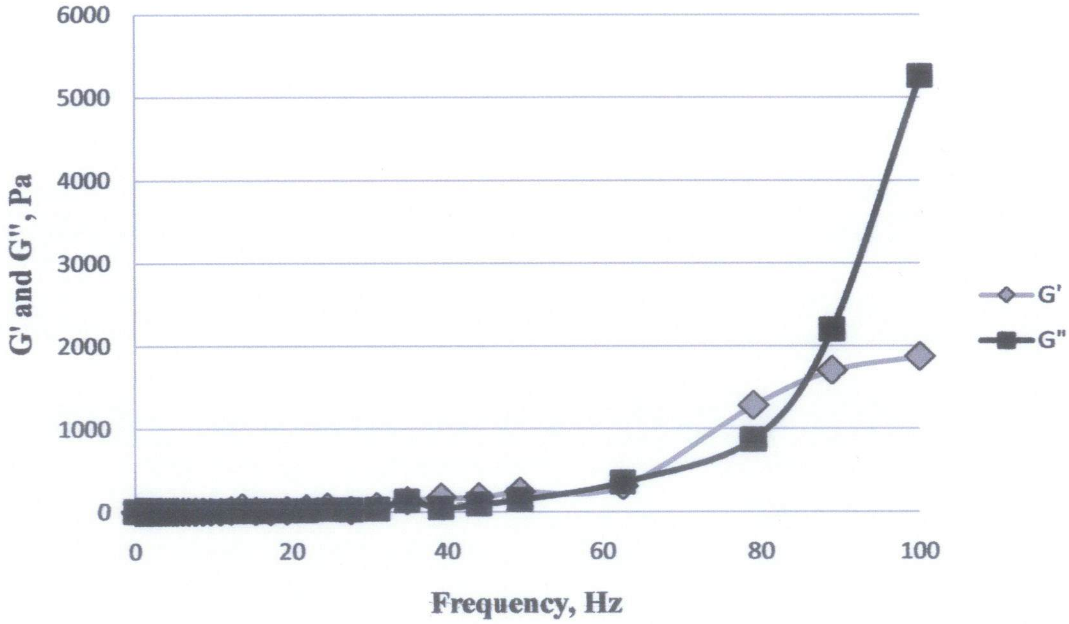


Figure 26: G' and G" profile of HPAM diluted solutions of 50ppm from 700pp m master solution

Meanwhile from **Figure 21** to **Figure 26** shown, the diluted solution 10ppm of 700ppm, 500ppm, and 300ppm had higher G" value than G' after one week left for hydration. The graph concluded that all diluted solutions from 700ppm represent more viscous properties than elastic properties. In comparison between all the diluted solutions in terms of viscous behavior, 10ppm solution behaves more towards viscous properties followed by 50ppm solution.

4.3.3 One month hydration period for 10, 20, and 50ppm dilutions

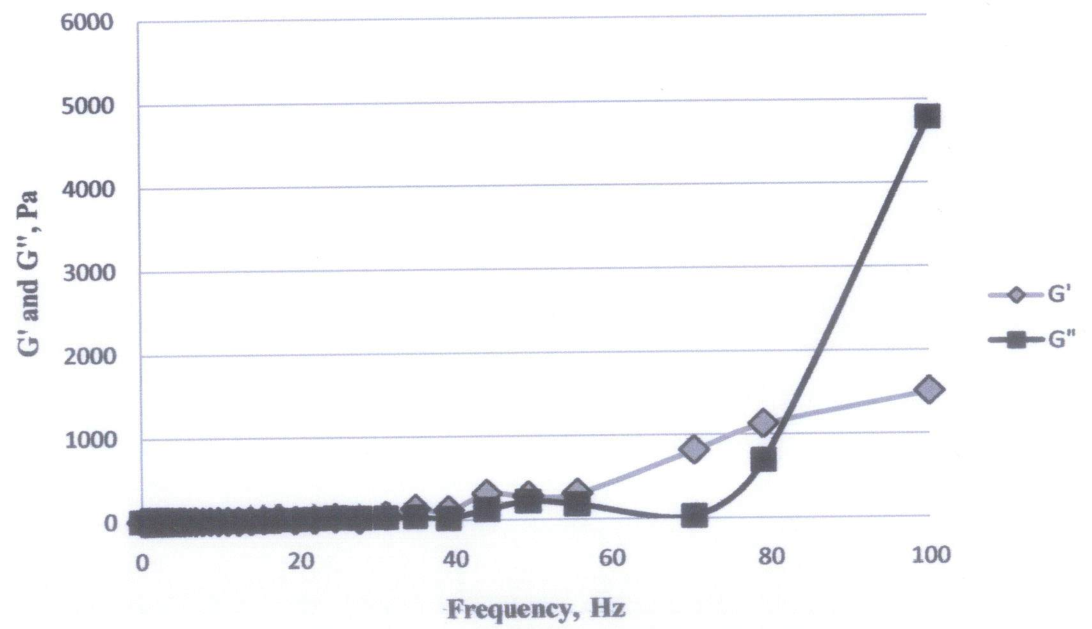


Figure 27: G' and G" profile of HPAM diluted solutions of 10ppm from 700ppm master solution

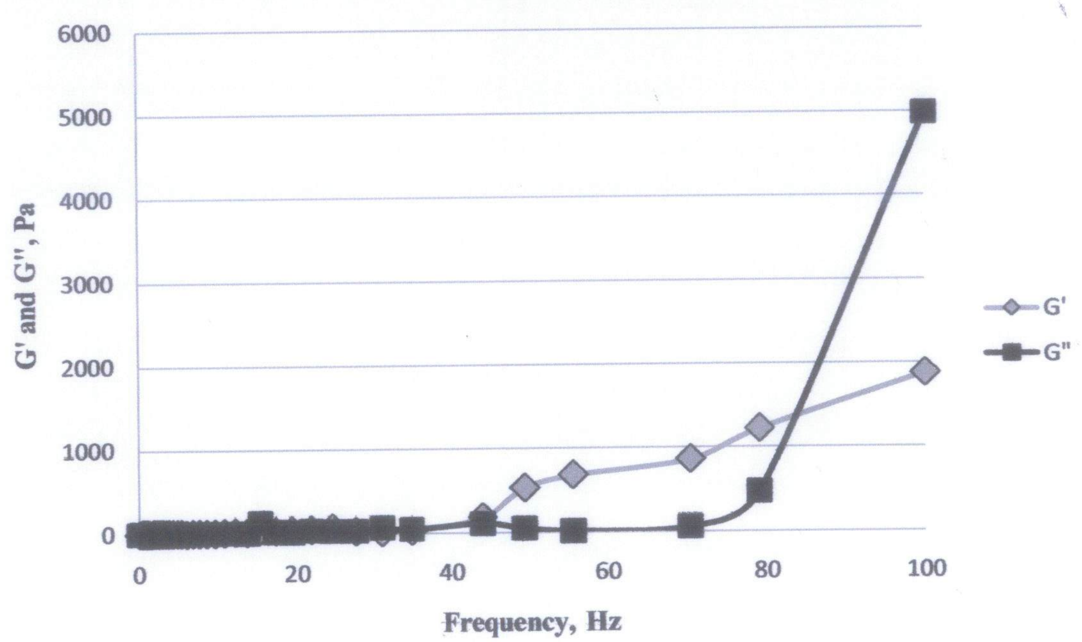


Figure 28: G' and G" profile of HPAM diluted solutions of 20ppm from 700ppm master solution

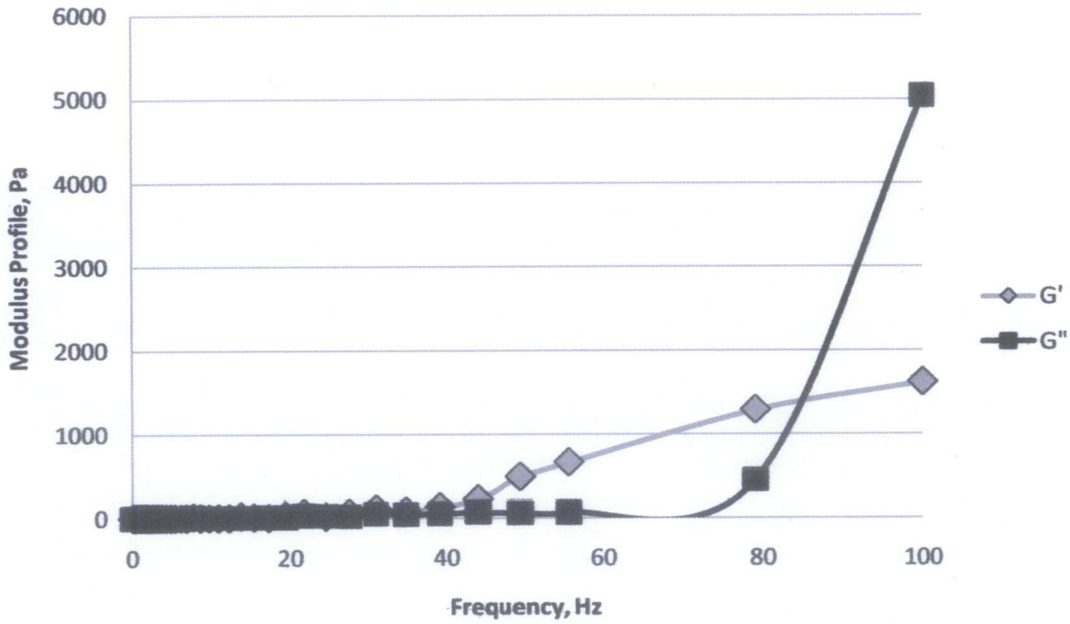


Figure 29: G' and G" profile of HPAM diluted solutions of 50ppm from 700ppm master solution

Based on the graph in **Figure** 27,

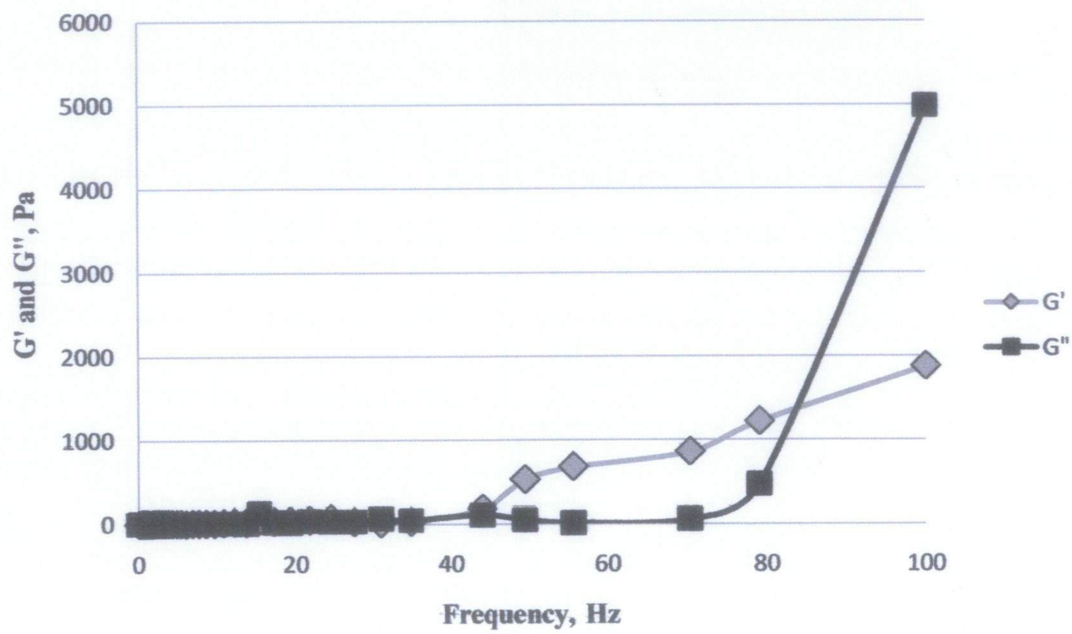


Figure 28, and **Figure 29**, the diluted solution 10ppm of 700ppm, 500ppm, and 300ppm had higher G" value than G' after one month left for hydration. The graph concluded

that all diluted solutions from 700ppm represent more viscous properties than elastic properties. In comparison between all the diluted solutions in terms of viscous behavior, 50ppm solution behaves more towards viscous properties followed by 10ppm solution.

4.4 Comparison between Elastic Modulus, G' and Viscous Modulus, G" of master solutions at various time of hydration

4.4.1 Elastic Modulus, G'

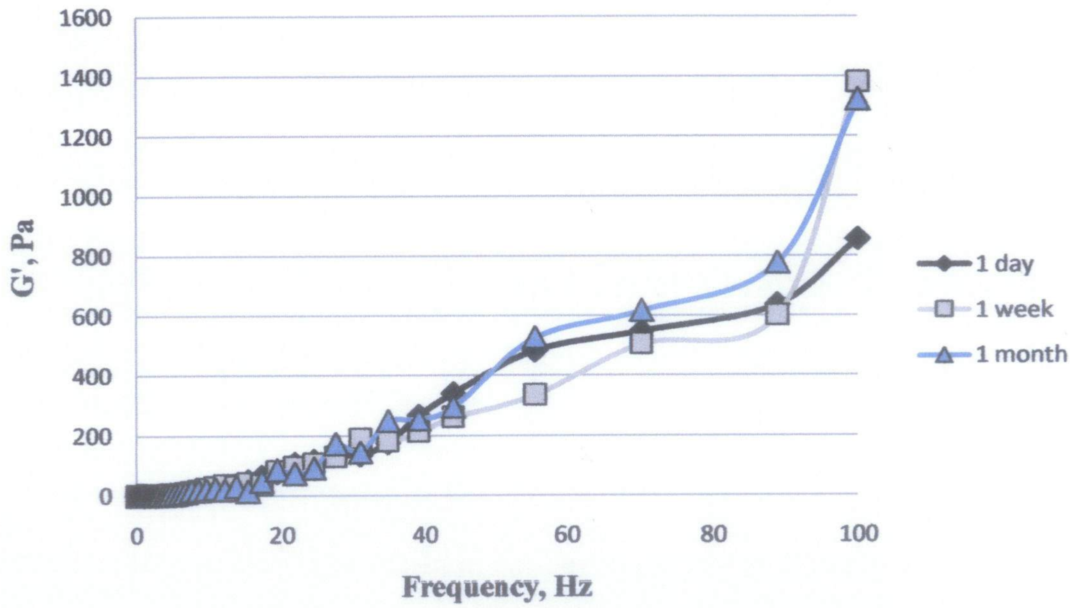


Figure 30: G' profile of HPAM master solutions of 700ppm at various time of hydration

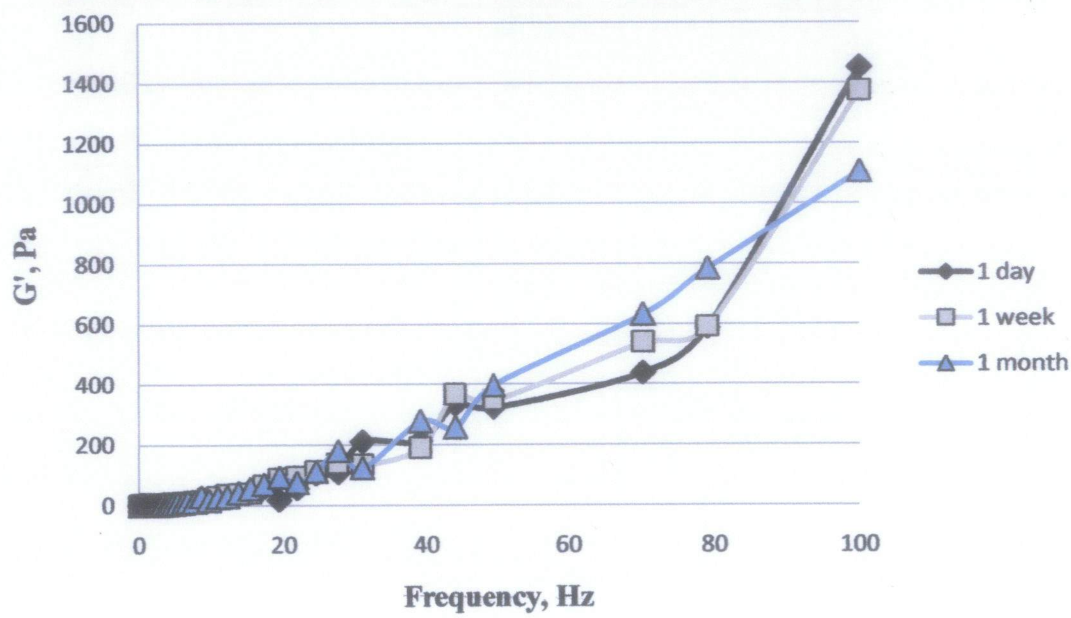


Figure 31: G' profile of HPAM master solutions of 500ppm at various time of hydration

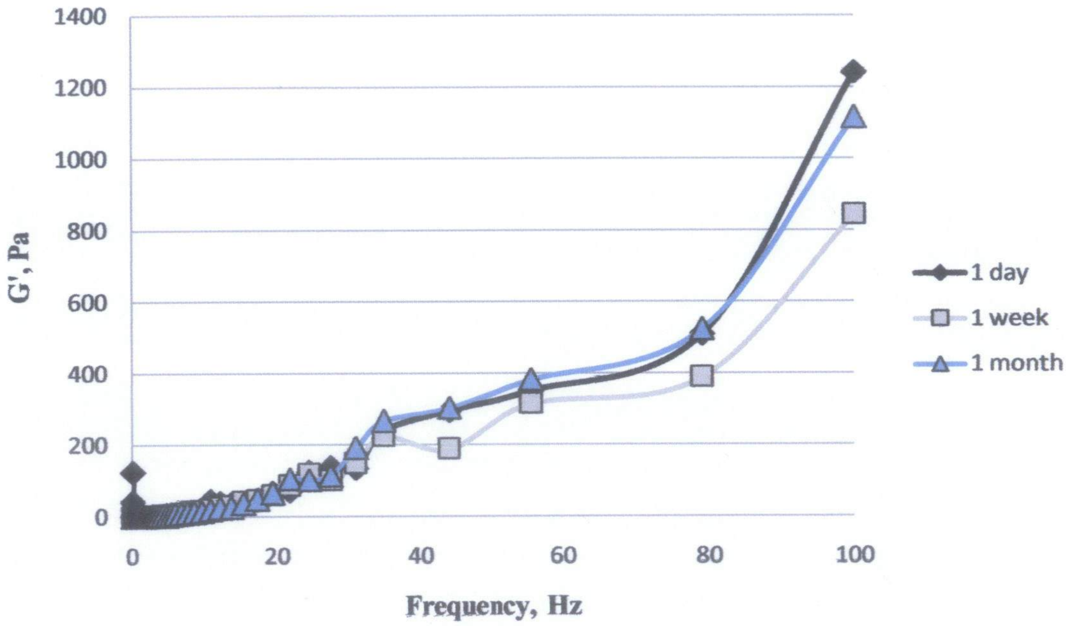


Figure 32: G' profile of HPAM master solutions of 300ppm at various time of hydration

Based on the results of elastic modulus with function of frequency at different time of hydration shown in Figure 30, Figure 31, and Figure 32, the HPAM solution of 500ppm solution shows higher G' value in 24 hours of hydration period compared to other master solutions. A high value of G' signifies the elastic properties of the polymer solutions.

Figure 30 represent the elastic modulus of 700ppm master solutions that showing increased elastic recovery after one week hydration. Meanwhile for Figure 31 and Figure 32, the master solution of 500ppm and 300ppm shows higher elasticity behavior after only 24 hours hydration time. The elastic modulus value for both master solutions decreased after one week hydration at a frequency of 100 Hz.

For overall results of G' profile, the elastic effect was dominated in the master solution of 500ppm which performs more elastic behavior than the other solutions.

4.4.2 Viscous Modulus, G”

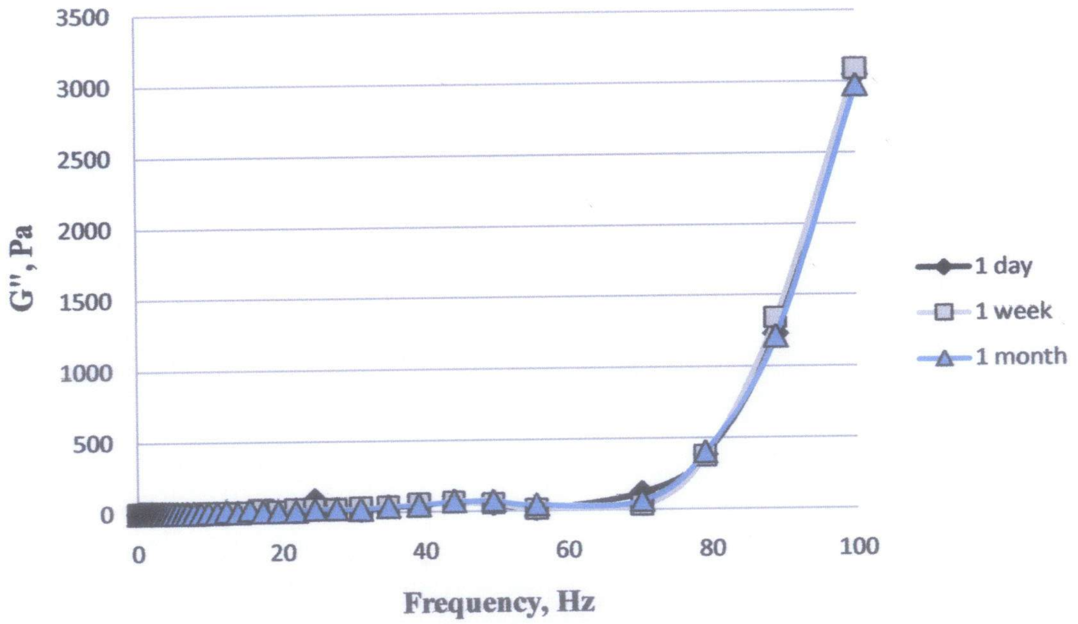


Figure 33: G” profile of HPAM solutions for 700ppm at various time of hydration

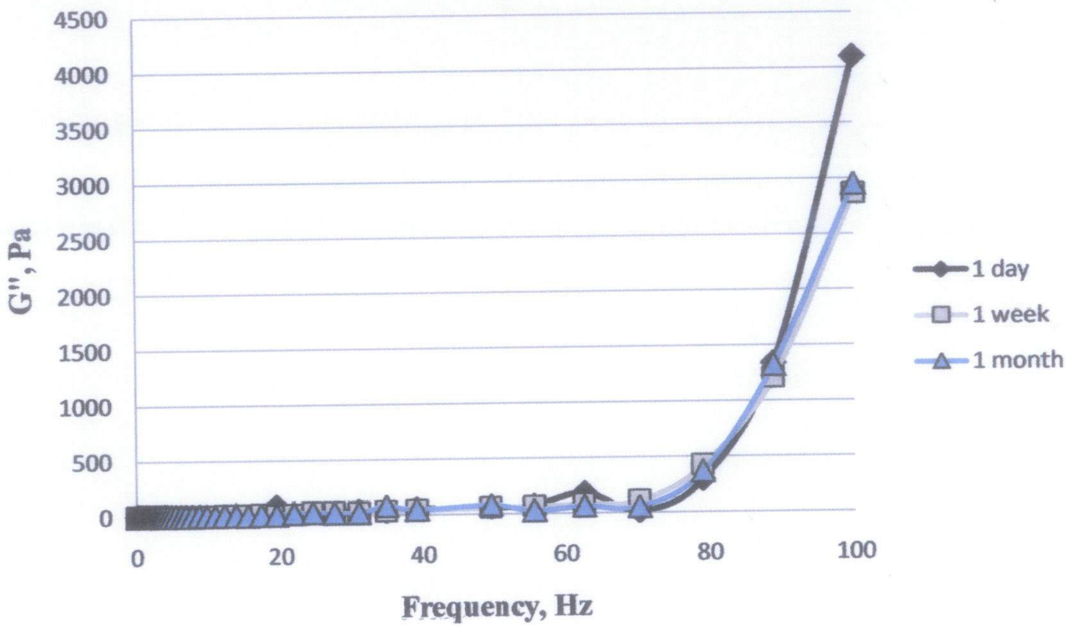


Figure 34: G” profile of HPAM solutions for 500ppm at various time of hydration

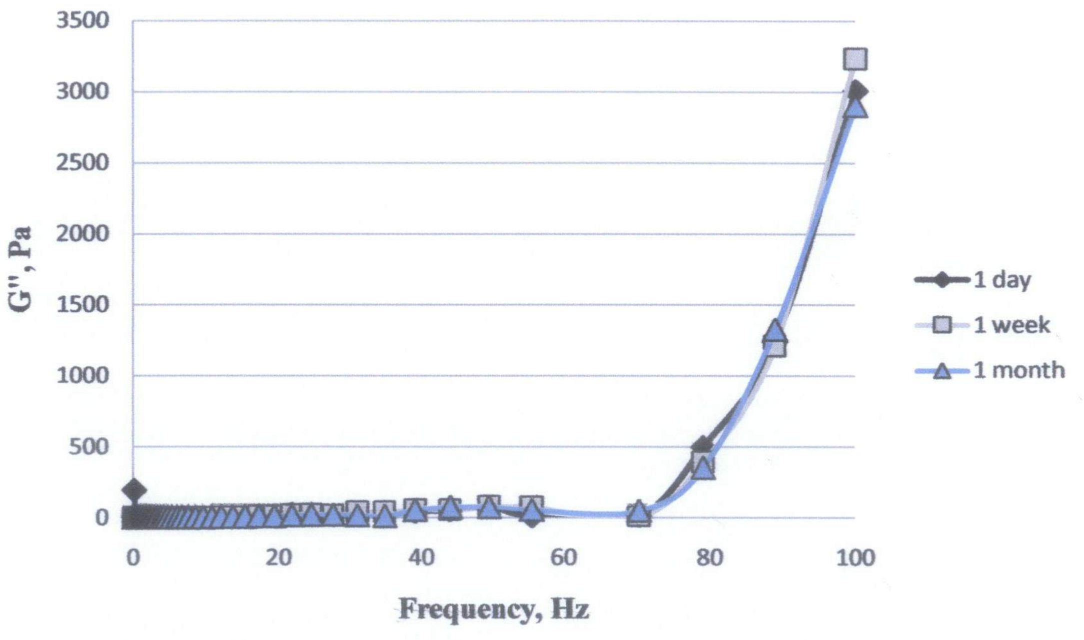


Figure 35: G" profile of HPAM solutions for 300ppm at various time of hydration

Based on the results of different time of hydration shown in Figure 33, Figure 34, and Figure 35, the HPAM solution of 700ppm and 300ppm solution shows higher G" value compared to 500ppm master solutions. A high value of G" signifies the viscous properties of the product.

Figure 33 shows that G" value has slightly increased after one week hydration period. After one month, the value of G" is then decreased back to the original value of one day hydration.

As seen from Figure 34, the viscous behavior for 500ppm solution exhibit more within 24 hours. After one week and one month left, it's G" properties has decreasing around 1 kPa. Meanwhile for 700ppm and 300ppm master solutions, the value of G" experience slightly increases after one week hydration at a frequency of 100 Hz.

From Figure 35, the viscosity characteristic rising approximately around 0.25 kPa after the solution was left for one week. After one month hydration, the value falls due to the degradation of the polymer solutions. Theoretically, most of the polymer solutions including HPAM will degrade after left more than 2 or 3 days.

CHAPTER 5: CONCLUSION & RECOMMENDATION

5.1 Conclusion

Investigation on the rheology behavior of HPAM to study the effect of viscous and elastic moduli subjected to:

- Master solution concentrations
- Diluted solution concentrations from master solutions
- Hydration time difference (one day, one week, and 1 month)

is accomplished. HPAM must maintain high viscosity to achieve good oil recovery and fluid phase flow transportation. Results indicated in Chapter 4 conclude that the study on the rheological properties through the viscous and elastic moduli of various master and diluted concentration in addition to the hydration time is accomplished. From the experiment, all master and dilute solution shows elastic and viscous behavior in different time of hydration.

Based on the findings of various master and dilute HPAM solution concentrations, it is proved that the hydration period affect the strength of the polymer solution. However, the results are not same as stated in the literature review studied by Zhao and his coworkers (Zhao et al., 2004). The results of their paper indicates that the higher the concentration, the higher the value of viscoelasticity. The data analysis on the rheology of HPAM (or ZETAG 4120) show that these polymer solutions increases the viscosity of injected water, reduces the mobility of water, and achieves a more stable displacement in predicting its performance and efficiency in EOR and flow enhancing process. All polymer solutions samples investigated behave **more towards viscous** solution than elastic. As an overall conclusion, concentrations and hydration period **does** affect on the viscoelasticity of HPAM.

5.2 Recommendation

In this project, the results of all master and dilute solutions from different time of storage were not as the same as the literature review as mentioned in Chapter 2. The experimental values cannot predict the theoretical experiment done by the researches of (Zhao et al., 2004) whom suggest that the viscoelasticity behaviour of HPAM solution increases with the increasing concentration of the polymer solution.

This phenomenon was affected mostly by the handling of the polymer solutions. The main effect is the temperature room effect since the temperature setting at 25°C sometimes is not very consistent as it keeps changing at around ±2% even in a closed room. This is because there is no solvent trap available at the moment during running the experiment. The solvent trap can distinguish the difference in temperature of the test samples. Plus, the results of the study can be extended by using different temperature setting on the rheometer using a solvent trap to evade other vibration or any temperature disturbance outside that may affect the test to run smoothly. Some of the master and diluted solution is left more than 24 hours hydration time due to time conflict with another commitment. Therefore, the accurate timing must be set up to ensure that the solution is left at exact 24 hours, one week and one month hydration period.

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APPENDICES

Graph Obtained from Bohlin Gemini II Rheometer at One Day Hydration

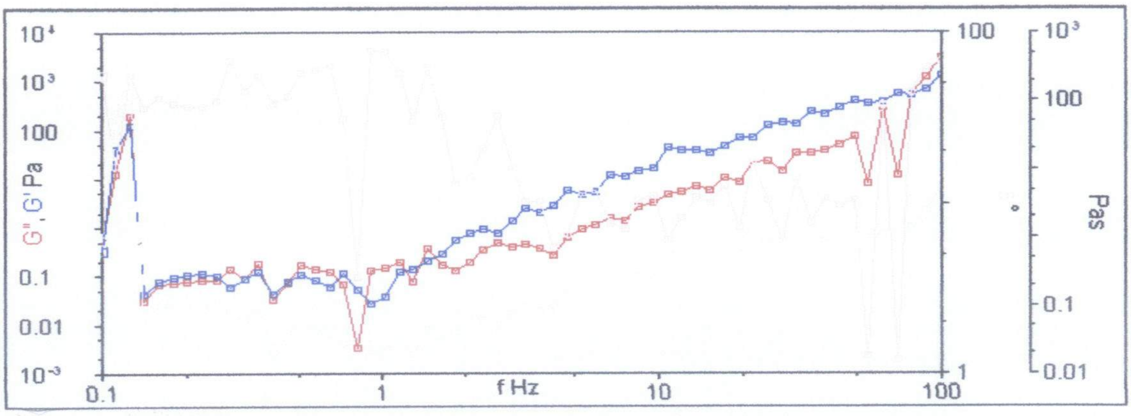

Figure 36: G' and G" profile vs frequency of 300ppm master solution

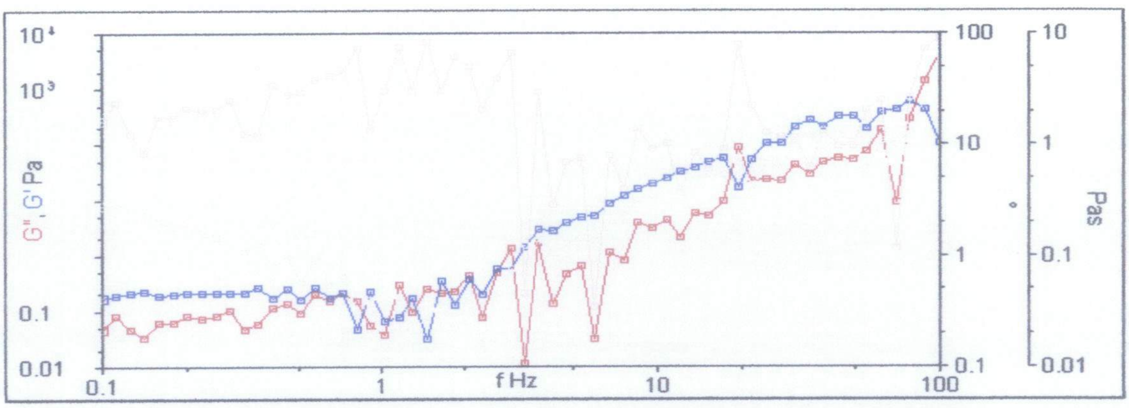

Figure 37: G' and G" profile vs frequency of 500ppm master solution

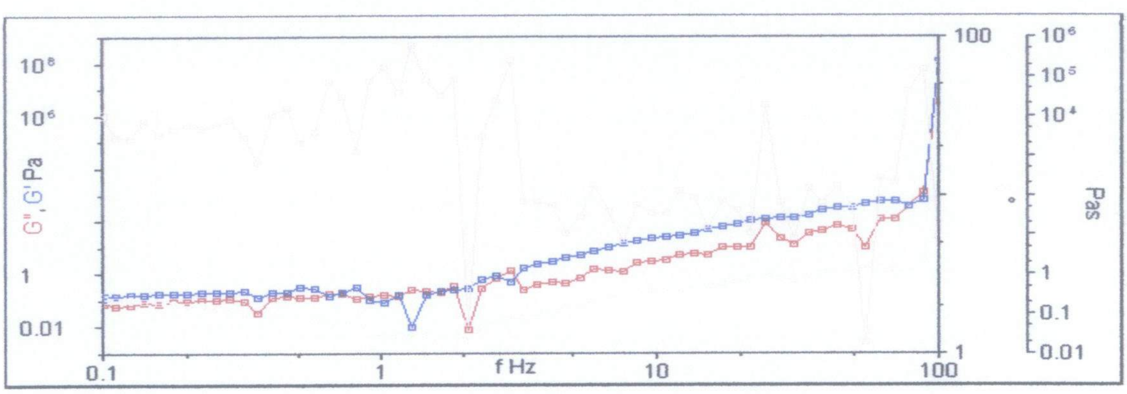

Figure 38: G' and G" profile vs frequency of 700ppm master solution

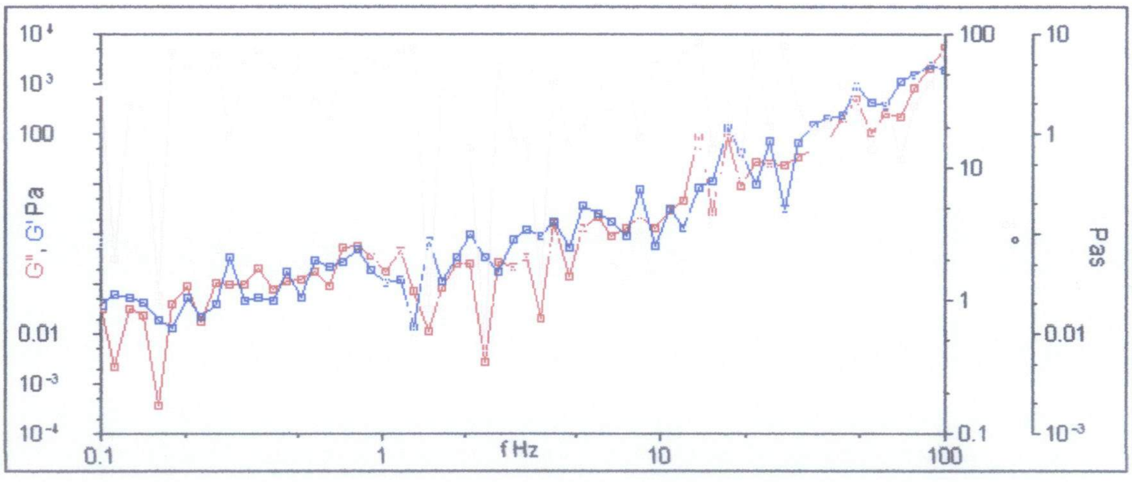

Figure 39: G' and G" profile of HPAM diluted solutions of 10ppm from 500ppm

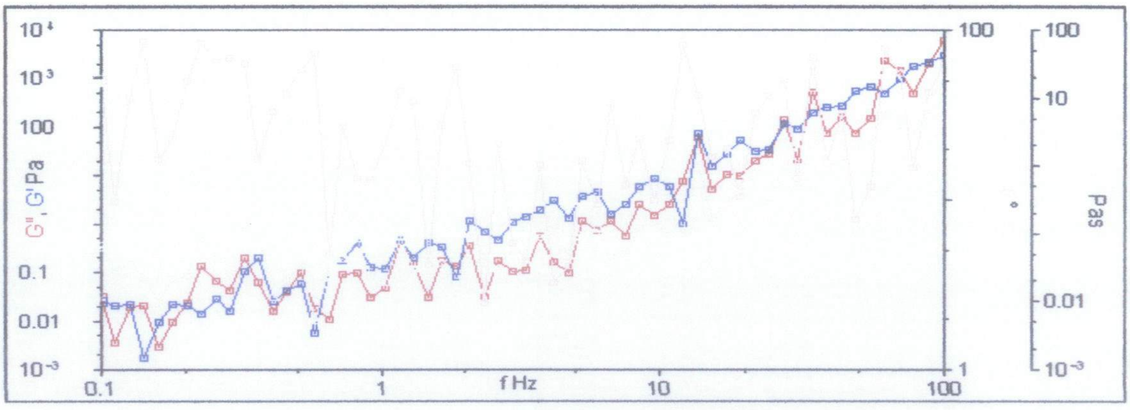

Figure 40: G' and G" profile of HPAM diluted solutions of 20ppm from 500ppm

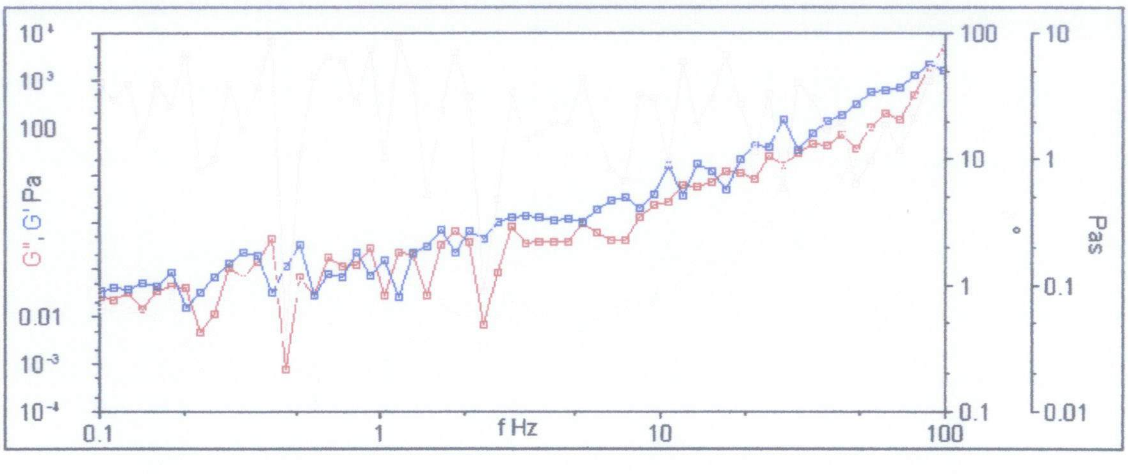

Figure 41: G' and G" profile of HPAM diluted solutions of 50ppm from 500ppm

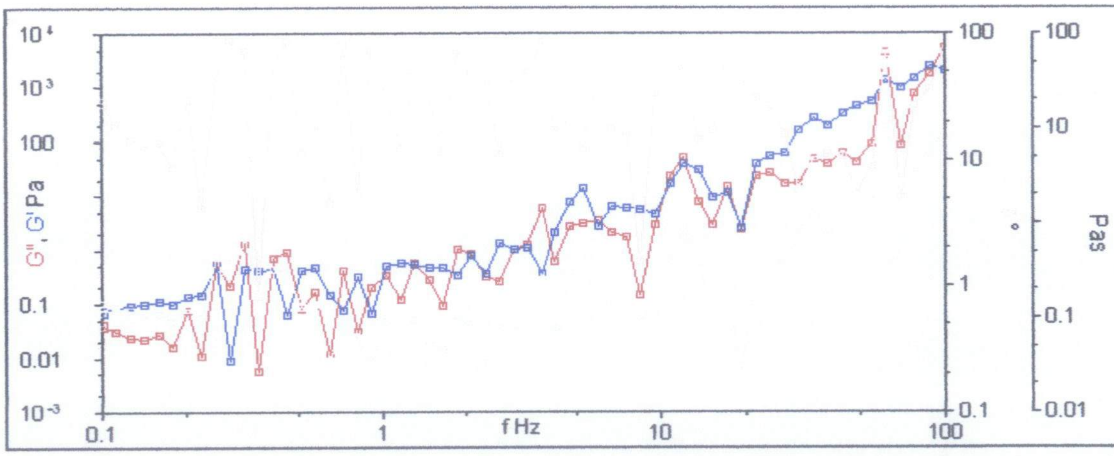

Figure 42: G' and G" profile of HPAM diluted solutions of 10ppm from 700ppm

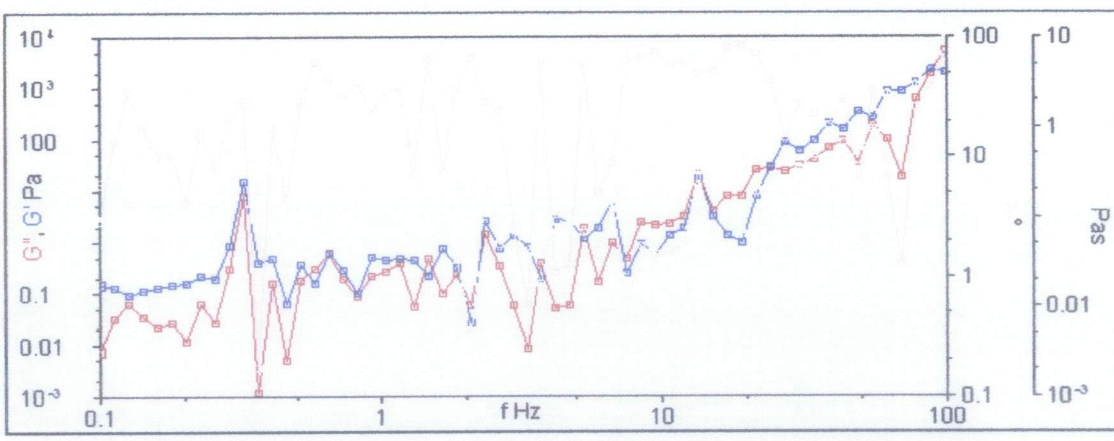

Figure 43: G' and G" profile of HPAM diluted solutions of 20ppm from 700ppm

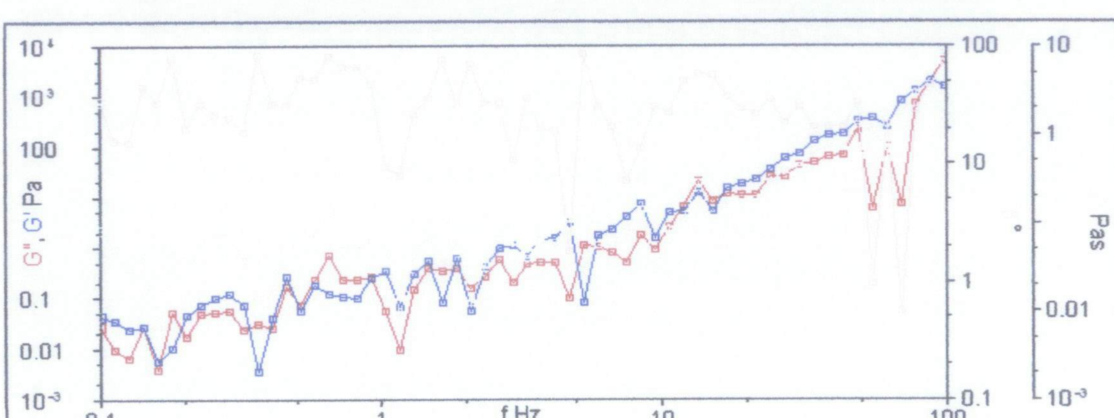

Figure 44: G' and G" profile of HPAM diluted solutions of 50ppm from 700ppm

Graph Obtained from Bohlin Gemini II Rheometer at One Week Hydration

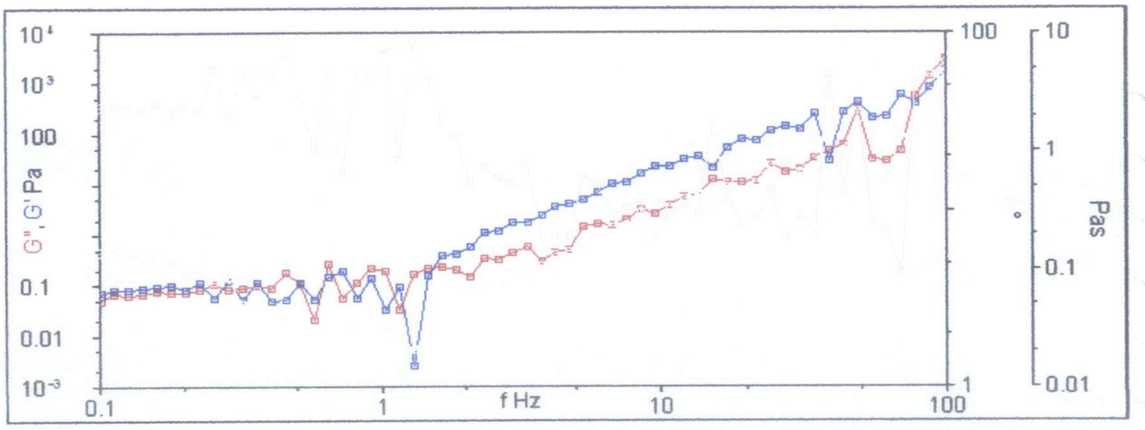

Figure 45: G' and G" profile vs frequency of 300ppm master solution

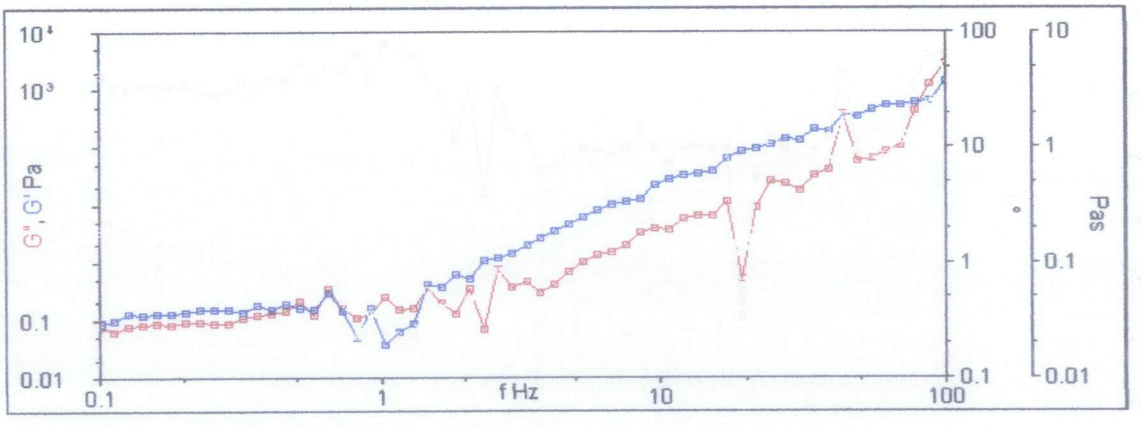

Figure 46: G' and G" profile vs frequency of 500ppm master solution

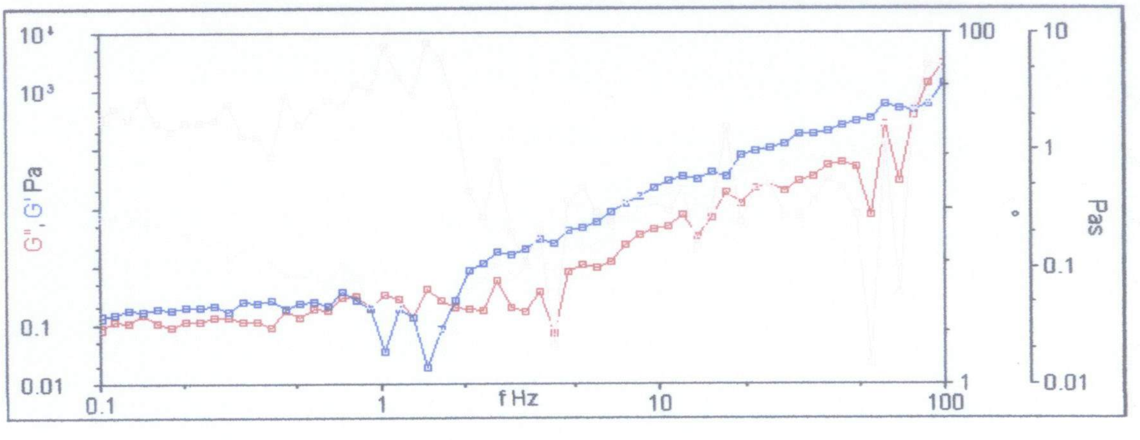

Figure 47: G' and G" profile vs frequency of 700ppm master solution

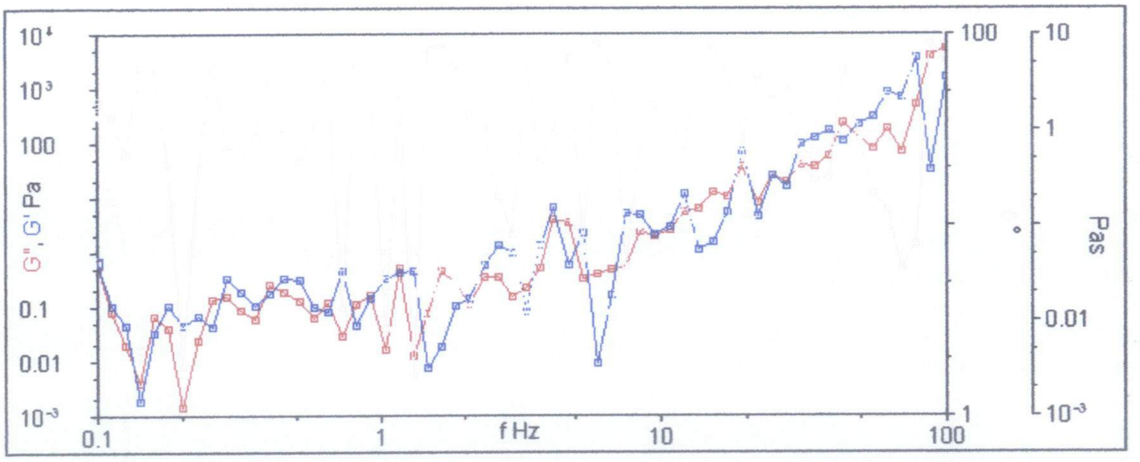

Figure 48: G' and G" profile of HPAM diluted solutions of 10ppm from 500ppm

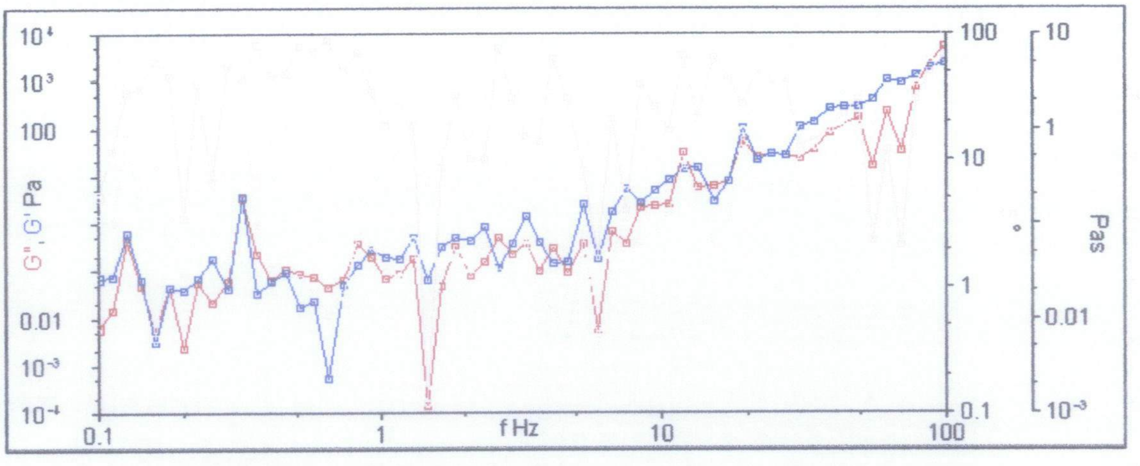

Figure 49: G' and G" profile of HPAM diluted solutions of 20ppm from 500ppm

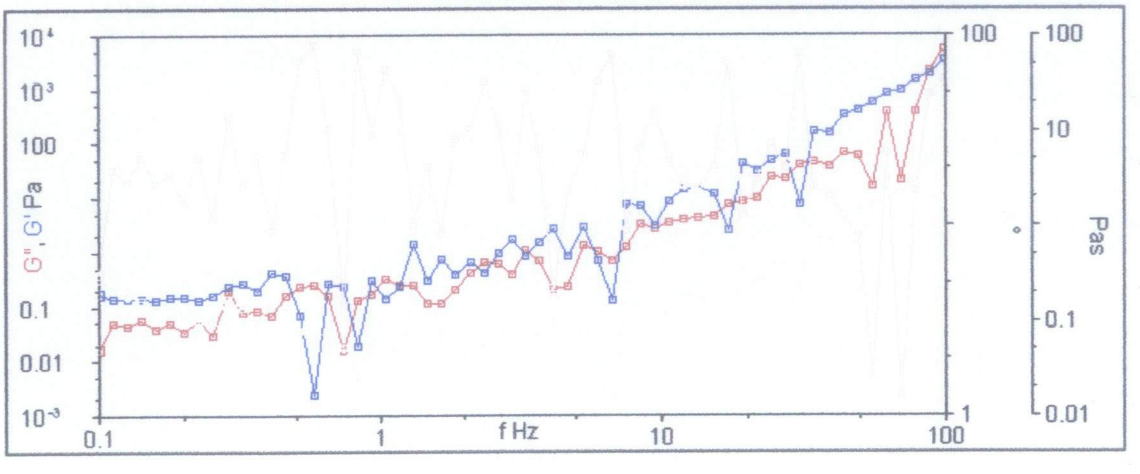

Figure 50: G' and G" profile of HPAM diluted solutions of 50ppm from 500ppm

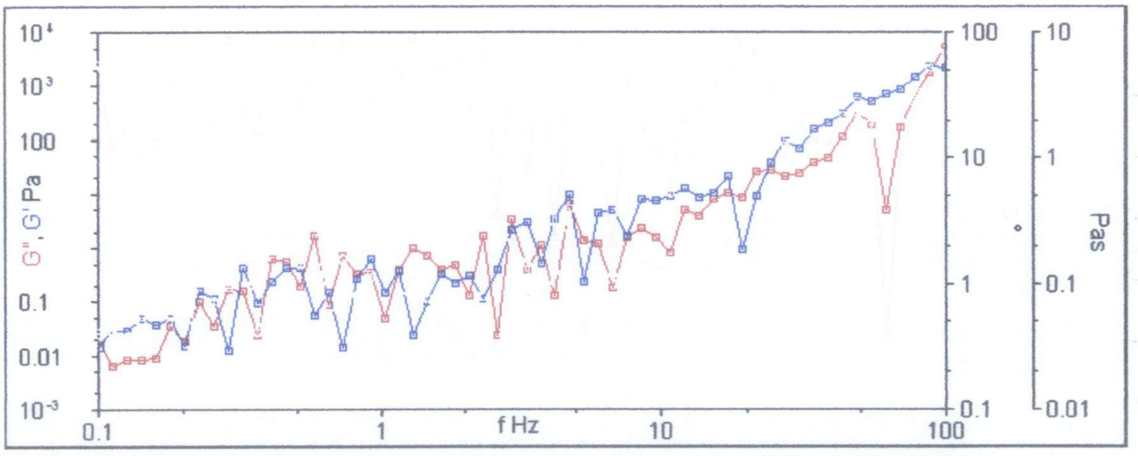

Figure 51: G' and G" profile of HPAM diluted solutions of 10ppm from 700ppm

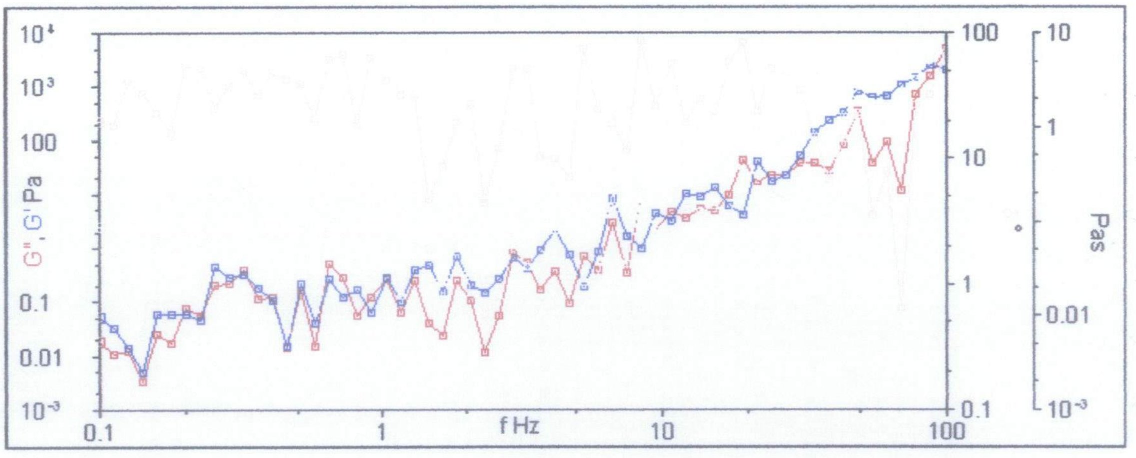

Figure 52: G' and G" profile of HPAM diluted solutions of 20ppm from 700ppm

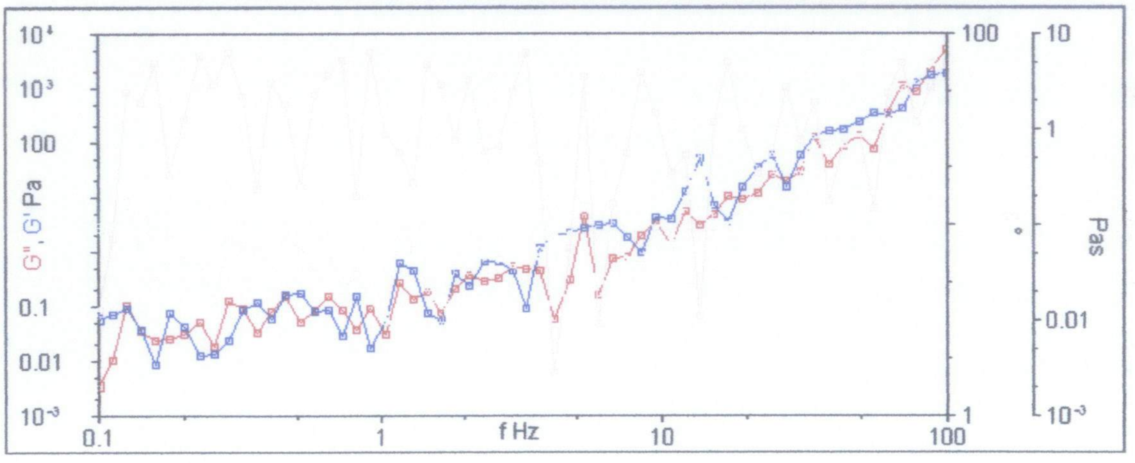

Figure 53: G' and G" profile of HPAM diluted solutions of 50ppm from 700ppm

Graph Obtained from Bohlin Gemini II Rheometer at One Month Hydration

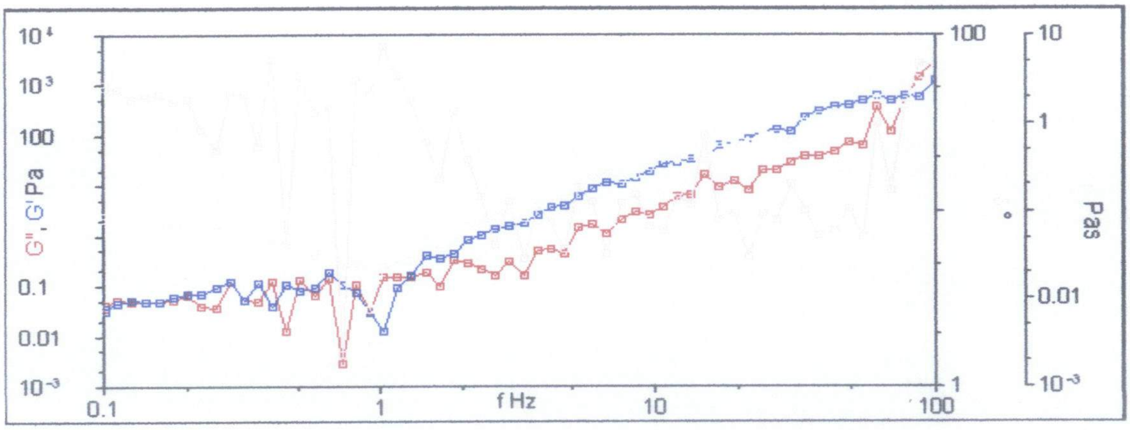

Figure 54: G' and G" profile vs frequency of 300ppm master solution

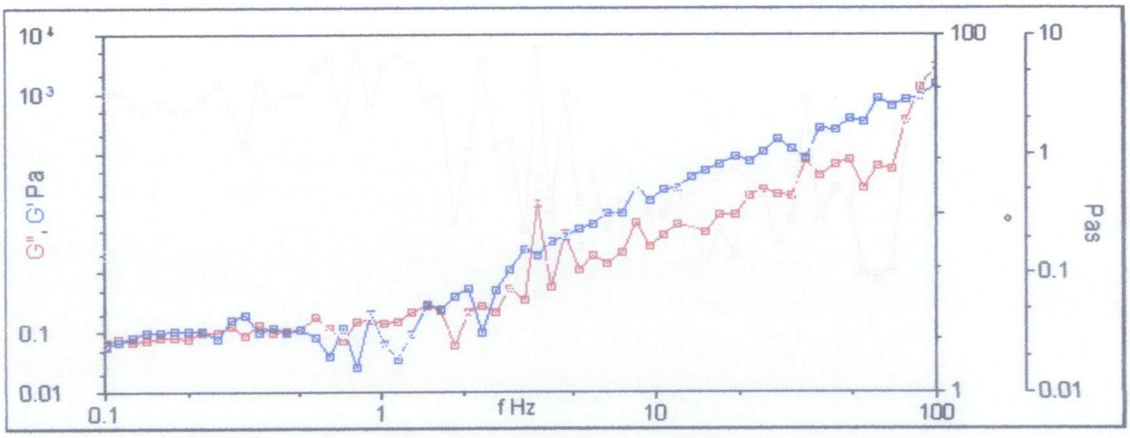

Figure 55: G' and G" profile vs frequency of 500ppm master solution

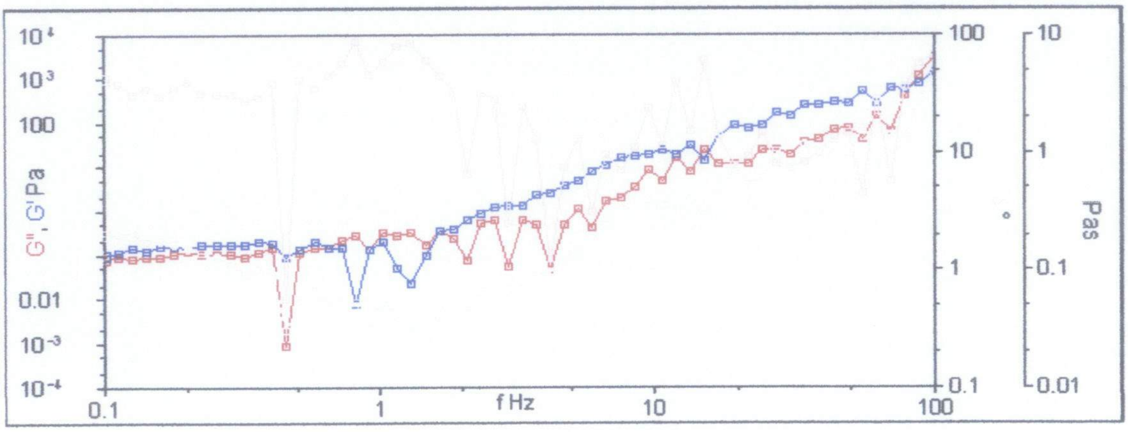

Figure 56: G' and G" profile vs frequency of 700ppm master solution

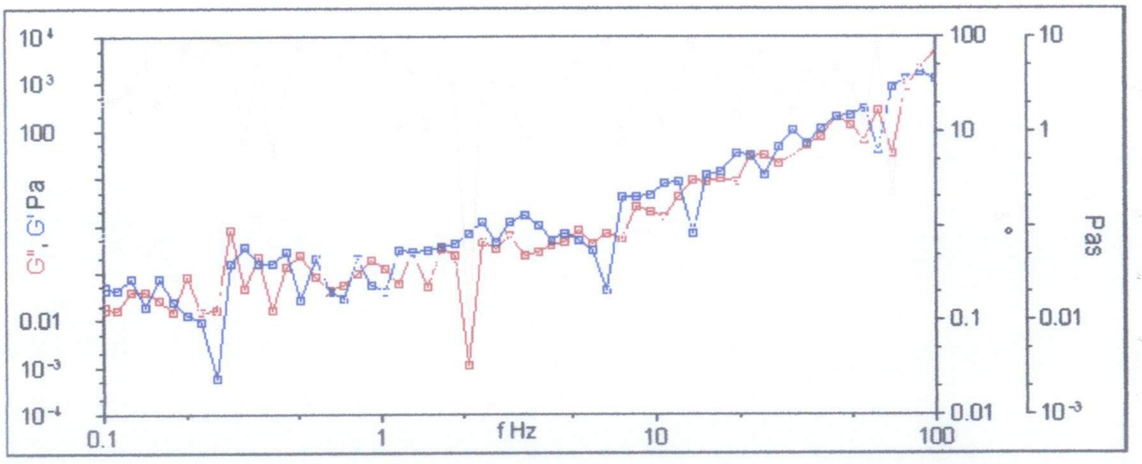

Figure 57: G' and G" profile of HPAM diluted solutions of 10ppm from 500ppm

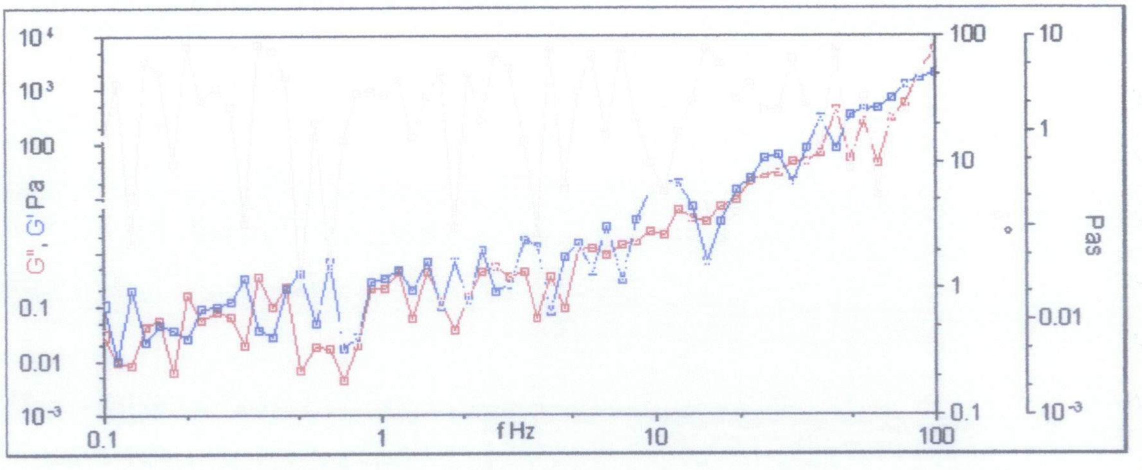

Figure 58: G' and G" profile of HPAM diluted solutions of 20ppm from 500ppm

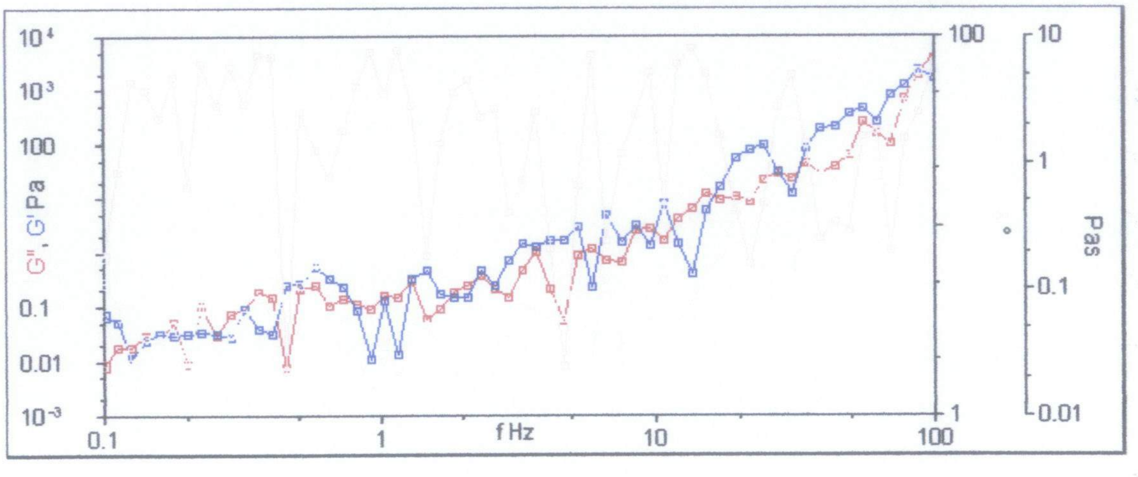

Figure 59: G' and G" profile of HPAM diluted solutions of 50ppm from 500ppm

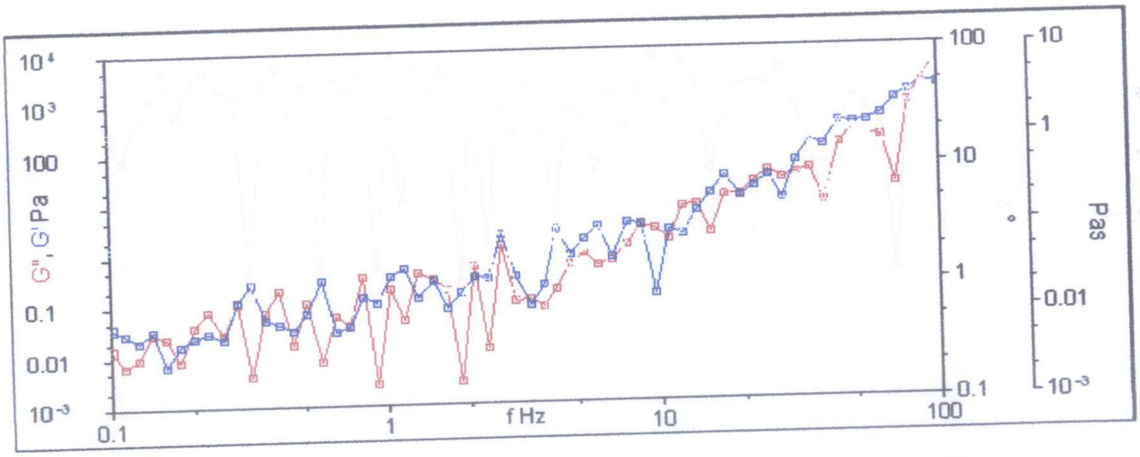

Figure 60: G' and G" profile of HPAM diluted solutions of 10ppm from 700ppm

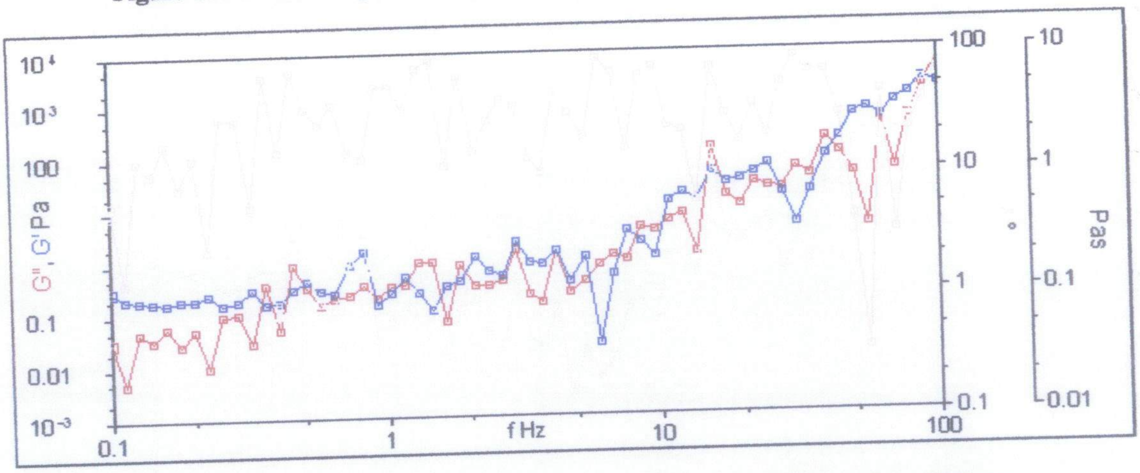

Figure 61: G' and G" profile of HPAM diluted solutions of 20ppm from 700ppm

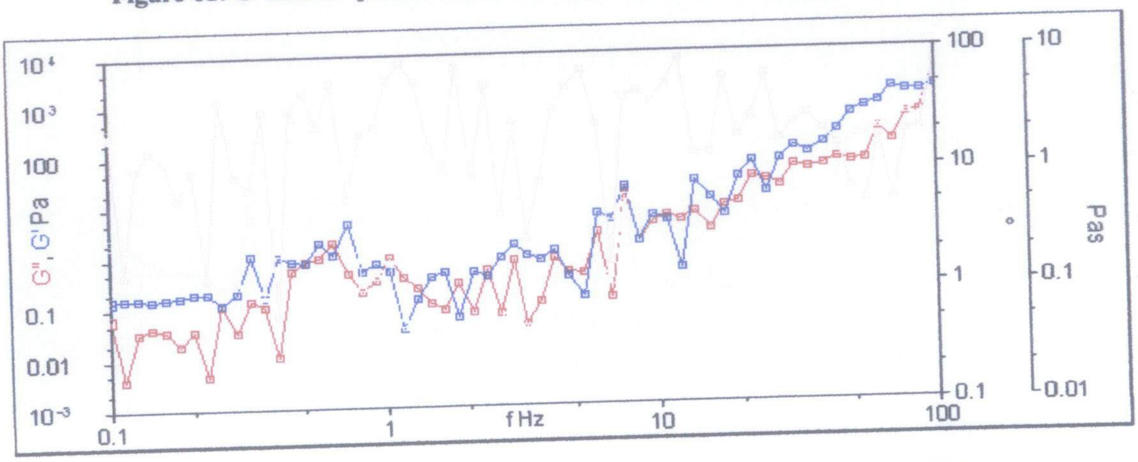

Figure 62: G' and G" profile of HPAM diluted solutions of 50ppm from 700ppm

Material Safety Data Sheet

PHPA L

I. PRODUCT AND COMPANY IDENTIFICATION

Product Name: PHPA L
Chemical Name: PARTIALLY HYDROLIZED POLYACRYLAMIDE
Chemical Family: SODIUM ACRYLATE & ACRYLAMIDE
Chemical Formula: PROPRIETARY
Synonyms: DRILL P, PHPA

Date Revised: 7/2010
CAS#: NDA

NFPA Properties: Health: 0 **Flammability:** 1 **Reactivity:** 0 **Contact:** 1

Supplier:

NOV FluidControl
4310 N Sam Houston Parkway East
Houston, Texas 77032 USA
Office: (713) 482-0500
Fax: (713) 482-0695
Company website: www.nov.com

Emergency Telephone Number:
CHEMTREC: 1-800-424-9300 or International +1-703-527-3887

II. HAZARDOUS INGREDIENTS/IDENTITY INFORMATION

Hazardous Components	TLV's (ACGIH) TWAPPM	TWA MG/M^3	STEL PPM	STEL MG/M^3	CAS#	OTHER LIMITS	%
1.							
2.							
3.							

III. PHYSICAL/CHEMICAL CHARACTERISTICS

Boiling Point °F: N/A
Specific Gravity: 0.8-1.0
Vapor Pressure: NDA
Percent Volatility: NDA
Vapor Density: N/A
Evaporation Rate: N/A
Solubility In Water: SOLUBLE
Melting Point °F: N/A

Color: WHITE
Odor: NONE
Appearance: VISCOUS LIQUID
pH:
Viscosity: N/A
Activity: 87-90 BY WT%
LC50: NDA
LD50: NDA

PHPA L — Material Safety Data Sheet

IV. FIRE & EXPLOSION HAZARD DATA

Extinguishing Agents: DRY CHEMICAL OR WATERSPRAY OR WATERFOG OR CO_2 OR FOAM OR SAND & EARTH

Flash Point °F: N/A

Flammable Limits: N/A LEL: N/A UEL: N/A

Special Firefighting Procedures: FIREFIGHTERS SHOULD WEAR PROPER PROTECTIVE EQUIPMENT AND SELF-CONTAINED (POSITIVE PRESSURE IF AVAILABLE) BREATHING APPARATUS WITH FULL FACEPIECE.

Unusual Fire & Explosion Hazards: NONE

Toxic Gases Produced: CARBON MONOXIDE, CARBON DIOXIDE

V. HEALTH HAZARD DATA

Routes of Entry: Inhalation: YES Skin: YES Ingestion: YES

Effects of Overexposure: DUST MAY IRRITATE EYES OR SKIN.

Toxicological Properties: NDA

Chronic & Acute Effects of Overexposure: NDA

Carcinogenicity: NTP: NO IARC Monographs: NO OSHA Regulated: NO

Emergency First Aid Procedures

Eyes: IMMEDIATELY FLUSH WITH LARGE QUANTITIES OF WATER FOR AT LEAST 15 MINUTES AND CALL A PHYSICIAN.

Skin Contact: FLUSH WITH LARGE AMOUNTS OF SOAP & WATER FOR 15 MINUTES.

Inhalation: REMOVE TO FRESH AIR, IF BREATHING IS DIFFICULT, GIVE OXYGEN AND CALL A PHYSICIAN.

Ingestion: GIVE LARGE AMOUNTS OF WATER AND CALL A PHYSICIAN.

VI. REACTIVITY DATA

Stability: STABLE

Hazardous Polymerization: WILL NOT OCCUR

Hazardous Decomposition Products: CARBON MONOXIDE, CARBON DIOXIDE, OXIDES, SMOKE, FUMES

Conditions To Avoid:

Incompatibility and Materials to Avoid: NDA

VII. SPILL & DISPOSAL PROCEDURES

Steps To Be Taken in Case Material is Released or Spilled --- Procedures For Clean – Up: WEAR SUITABLE PROTECTIVE CLOTHING. SWEEP UP WITH CLEAN EQUIPMENT AND PLACE IN APPROPRIATE CONTAINER. HYDRATING THIS MATERIAL WILL PRODUCE AN EXTREMELY SLICK/SLIPPERY SURFACE.
Waste Disposal Method: DISPOSE OF IN ACCORDANCE WITH ALL APPLICABLE FEDERAL, STATE AND LOCAL REGULATIONS.
Precautions To Be Taken In Handling & Storage: NONE

VIII. PROTECTIVE EQUIPMENT

Ventilation Type Required: MECHANICAL
Protective Gloves: RUBBER OR PLASTIC
Respiratory Protection: WEAR A NIOSH APPROVED MASK IF CONCENTRATION IS TO EXCEED TLV
Other Protective Equipment:
Comments:

IX. REGULATORY & TRANSPORTATION INFORMATION

US DOT Proper Shipping Name: "OIL – WELL TREATING COMPOUND"
US DOT Hazard Class:
ID Number:
Unregulated By DOT:
Special Transportation Note:
Labels Required:

DOT ID Number:
Freight Classification:
Regulated by DOT: NO

DISCLAIMER:
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